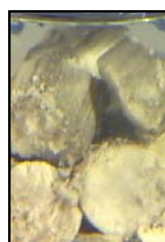


# Tank 25F, 28F and 44F Saltcake Samples



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Savannah River National Laboratory, Aiken, SC 29808  
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**Savannah River/Hanford/Idaho Technical Exchange, Atlanta, Georgia**  
**Wednesday, October 10, 2007**

**WSRC-STI-2007-00526-S**

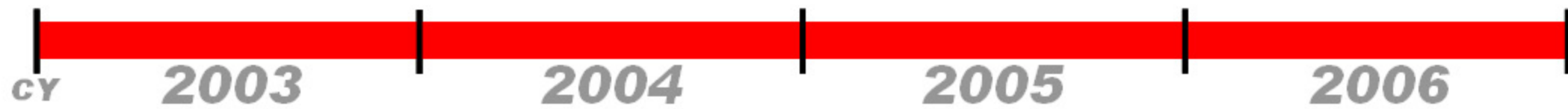
# Presentation Outline

- **Objectives and Timeline** of Saltcake Core Sampling
- **Tank 25F:** full draining and dissolution test
  - **Segment Analysis** for vertical variation and analyte correlations
  - **Composite Draining** for Interstitial Liquid removal
  - **Dissolution Test** permitted analysis of three batches and heel
- **Tank 28F:** update from previous technical exchange
  - **Segment Analysis** of wet samples showed correlation of solubles
- **Tank 44F:** received for future processing
  - **Visual Inspection** showed obvious variation

# Background and Objectives

- **16.5 million gallons of High Level Waste saltcake at SRS.**
  - Characterization difficult... heterogeneous material that can't be mixed.
- **Characterization of saltcake needed to reduce processing risk, project composition of dissolved salt batches.**
  - Actinides, Cs-137, Sr-90 and other important species
  - Critical process feed requirements (for caustic-side solvent extraction)
  - Saltstone Waste Acceptance Criteria (WAC) for other radionuclides
  - Residual heel properties
- **Provide basis for determining number and type of samples for future tanks**

# Saltcake Sampling Timeline





# Saltcake Sampling Timeline



- **Pre 2003:** augering, 3" and 8" cups into surface/wells



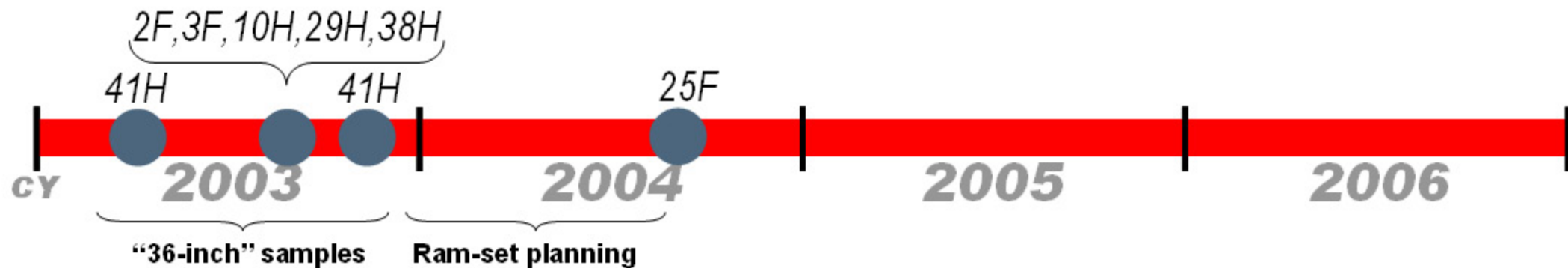
# Saltcake Sampling Timeline



- **Pre 2003:** augering, 3" and 8" cups into surface/wells
- **2003:** up to 36" into surface, seven cores from six tanks



# Saltcake Sampling Timeline

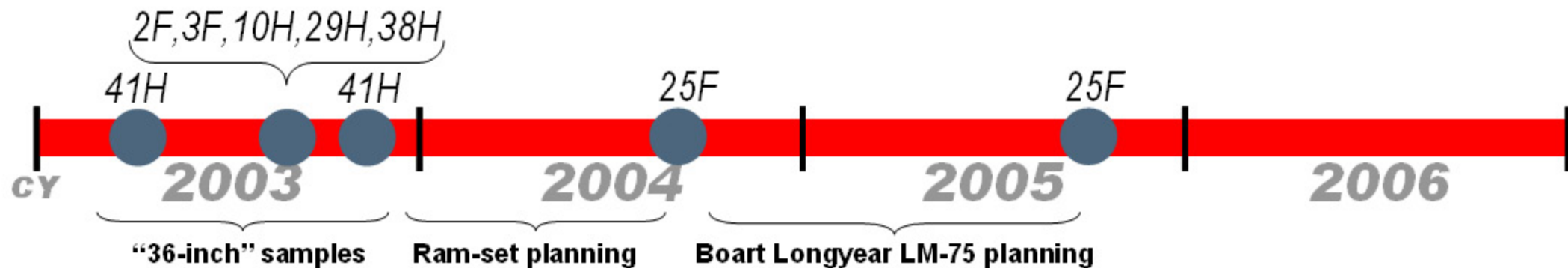


- **Pre 2003:** augering, 3" and 8" cups into surface/wells
- **2003:** up to 36" into surface, seven cores from six tanks
- **2004:** ram-set refusal in Tank 25F





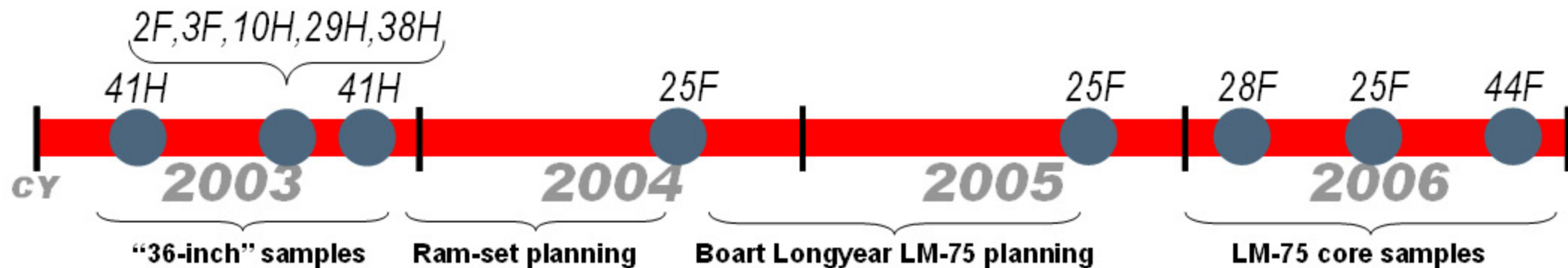
# Saltcake Sampling Timeline



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- **2003:** up to 36" into surface, seven cores from six tanks
- **2004:** ram-set refusal in Tank 25F
- **2005:** initial Tank 25F attempt w/ LM-75, little sample



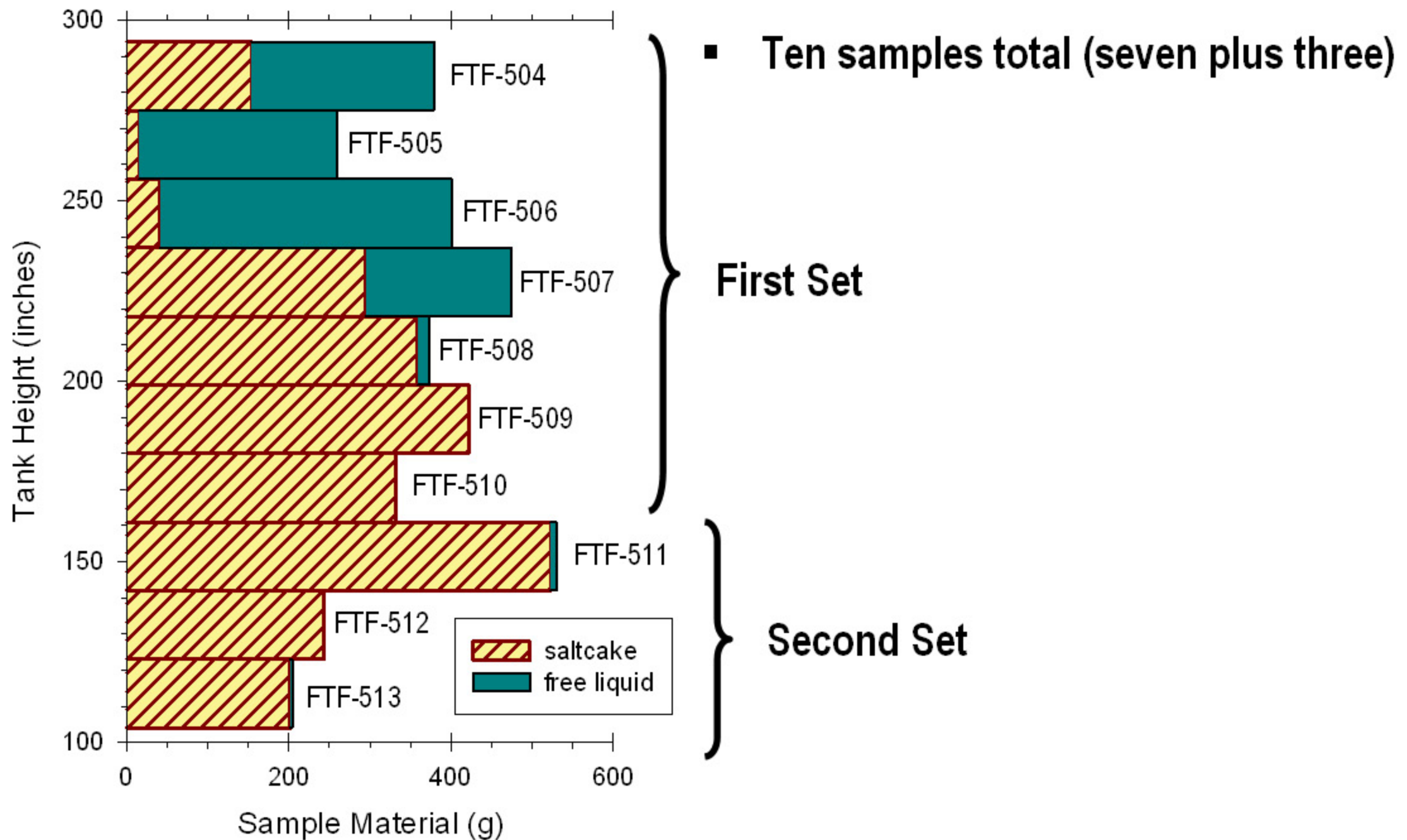
# Saltcake Sampling Timeline



- **Pre 2003:** augering, 3" and 8" cups into surface/wells
- **2003:** up to 36" into surface, seven cores from six tanks
- **2004:** ram-set refusal in Tank 25F
- **2005:** initial Tank 25F attempt w/ LM-75, little sample
- **2006:** samples in this report, Tanks 28F, 25F, and 44F



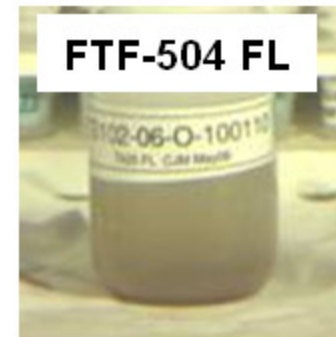
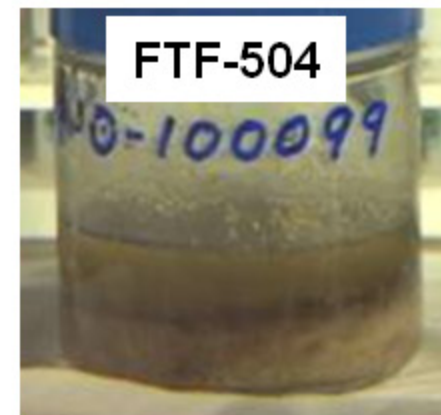
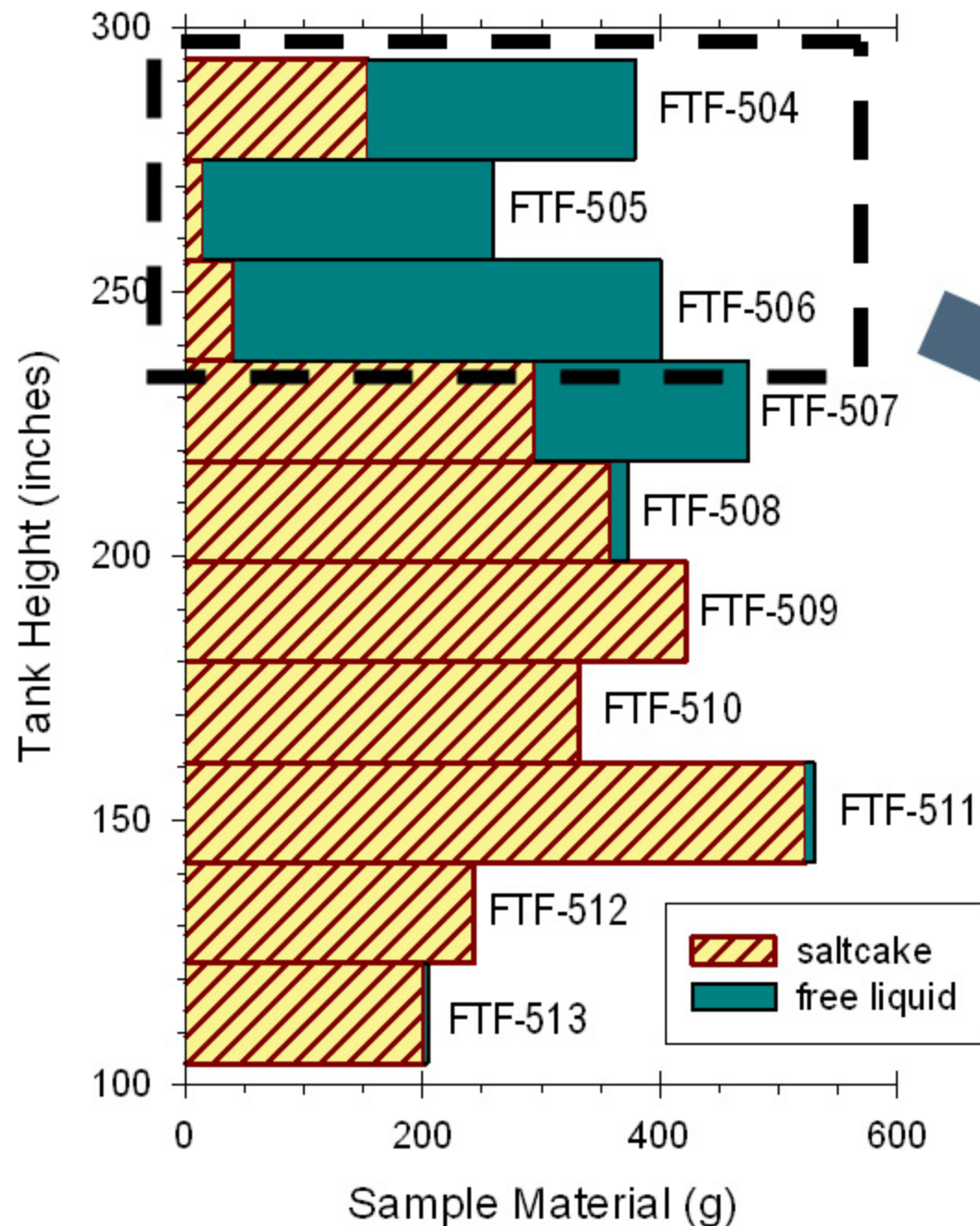
# Tank 25F Saltcake Core Sample Profile



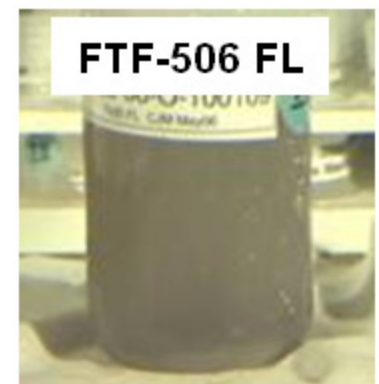
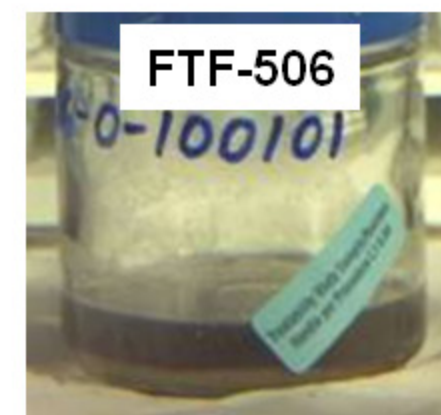
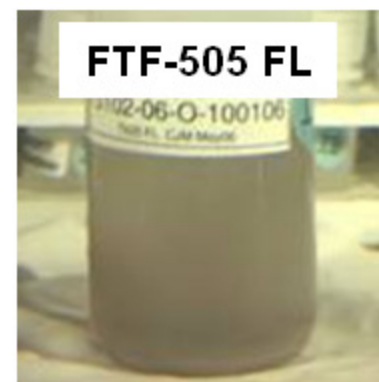


# Tank 25F Saltcake Core Sample Profile

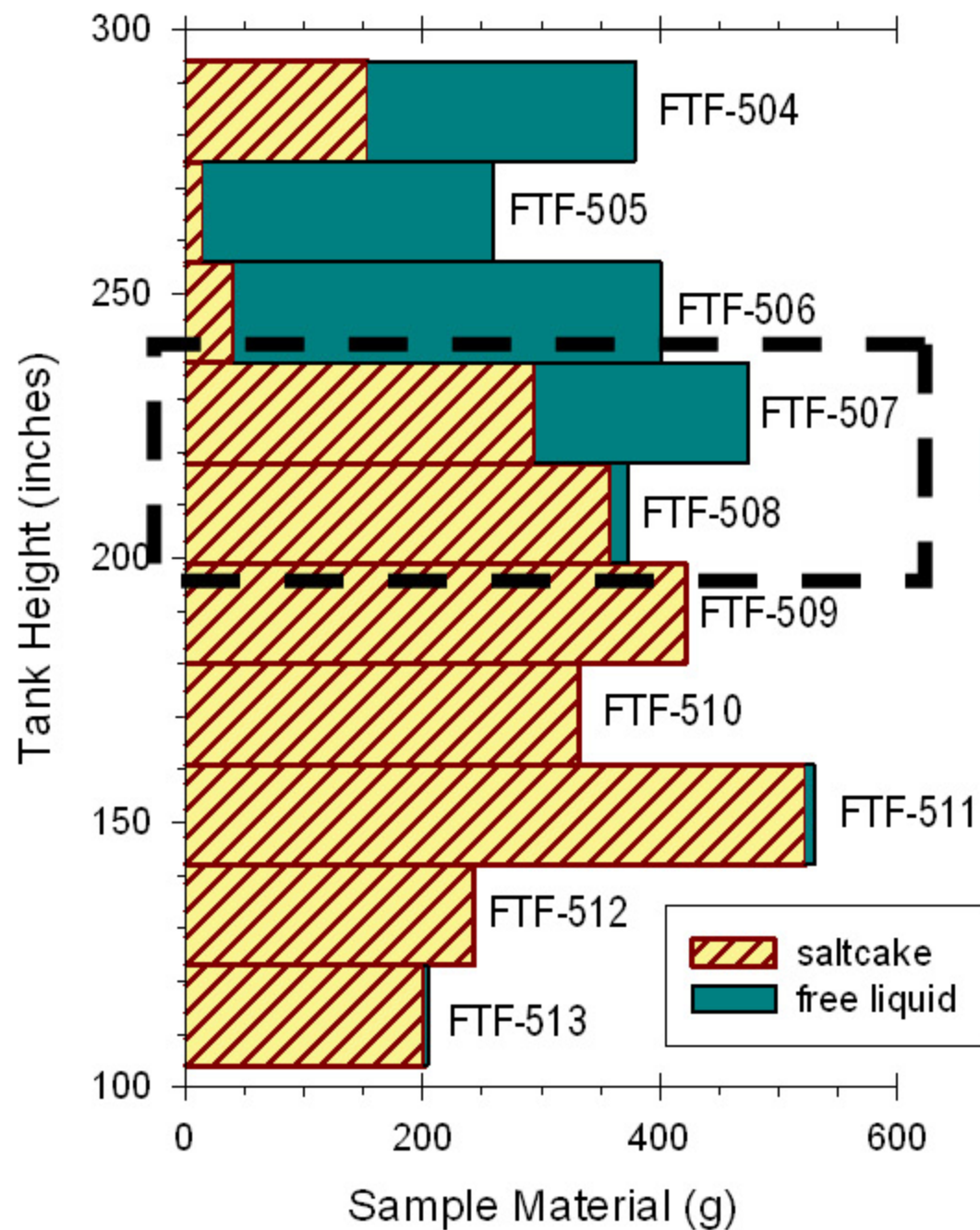
- Ten samples total (seven plus three)
- Top three samples, wet with Free Liquid



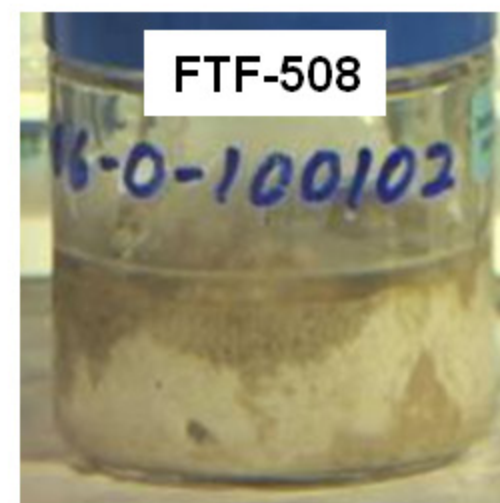
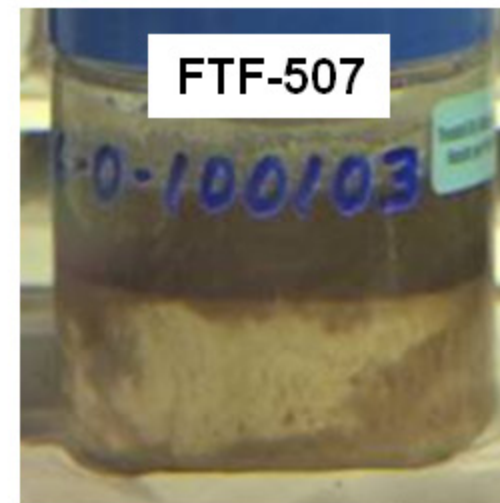
FTF-505  
(no jar)



# Tank 25F Saltcake Core Sample Profile

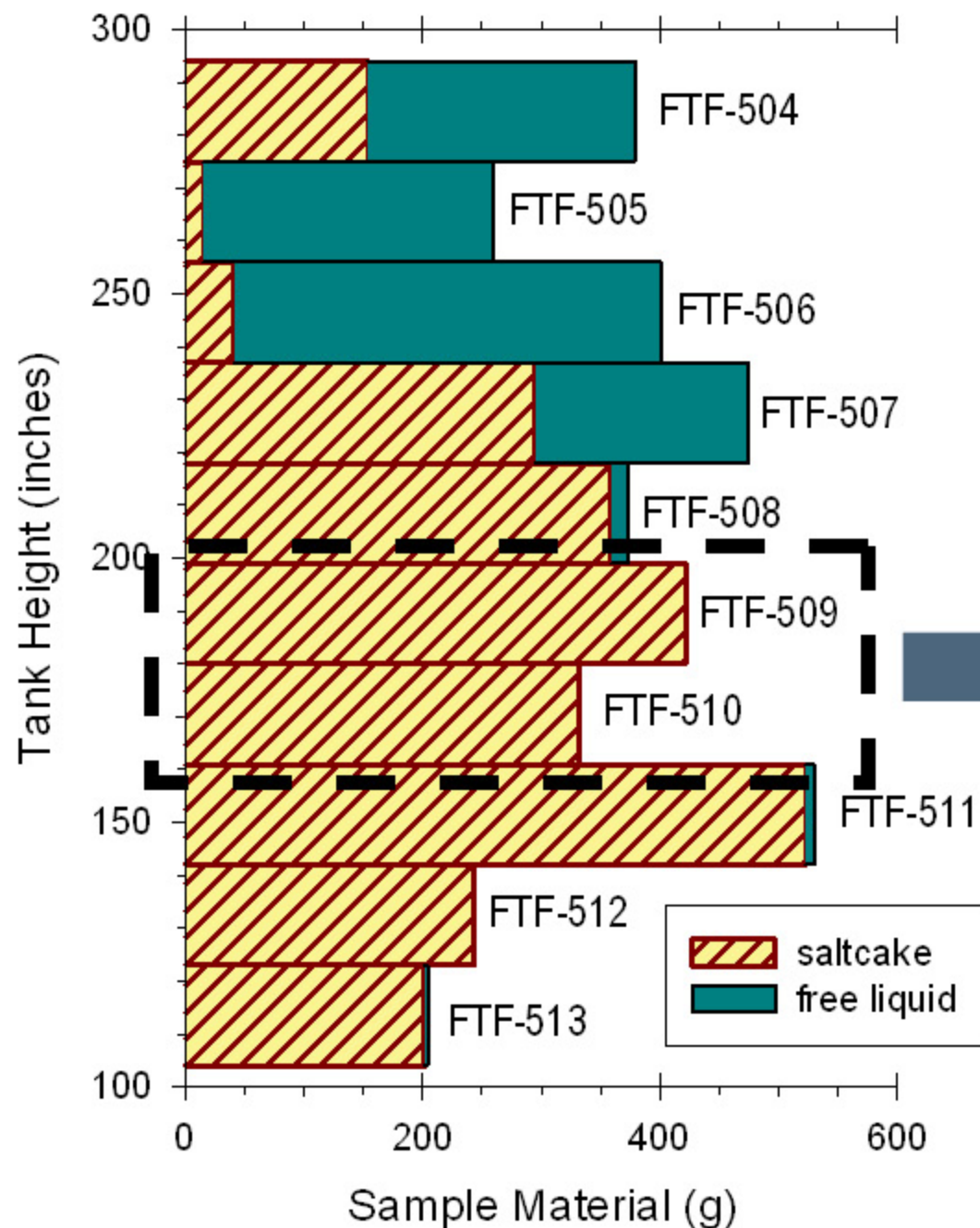


- Ten samples total (seven plus three)
- Top three samples, wet with Free Liquid
- Next two samples, soft and moldable

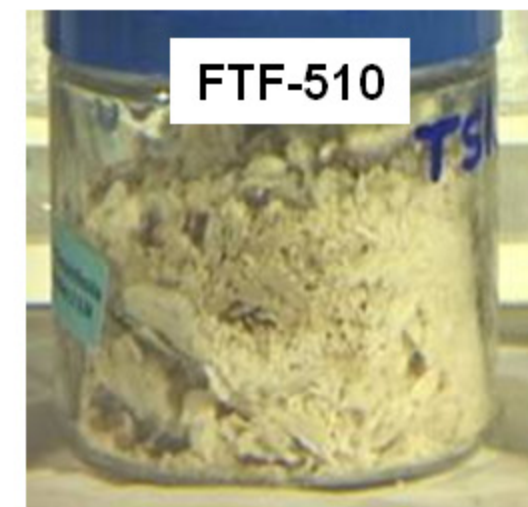
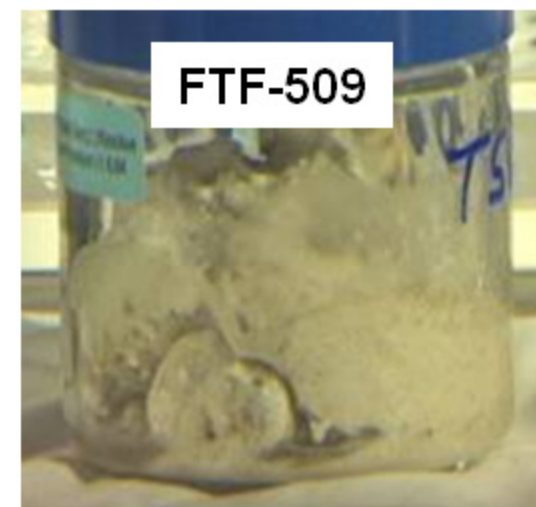




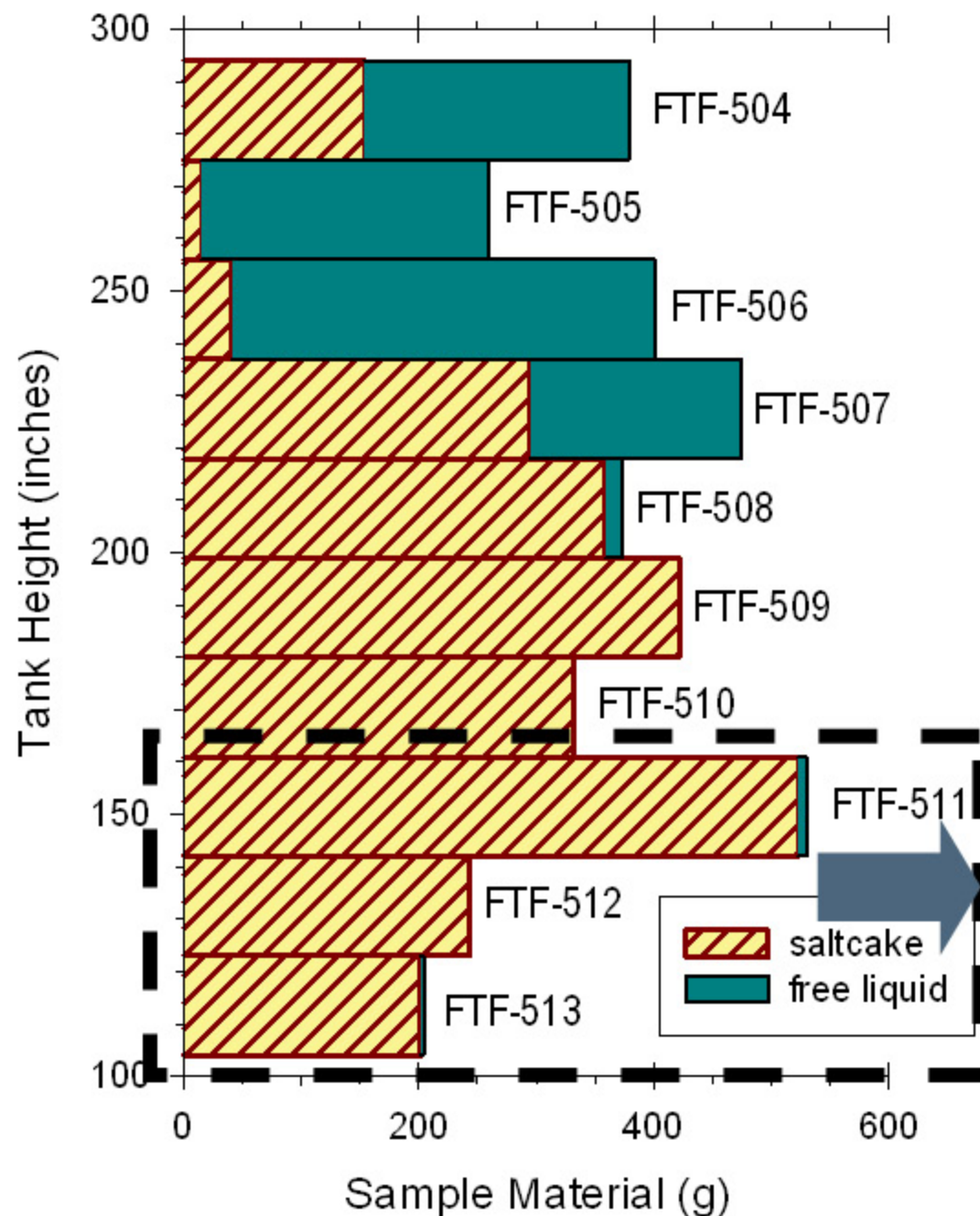
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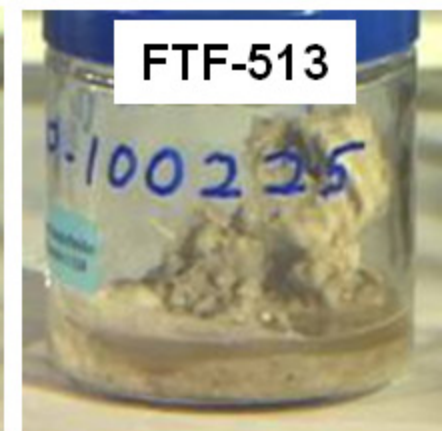
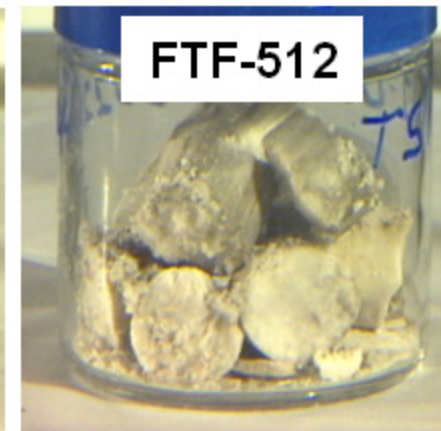
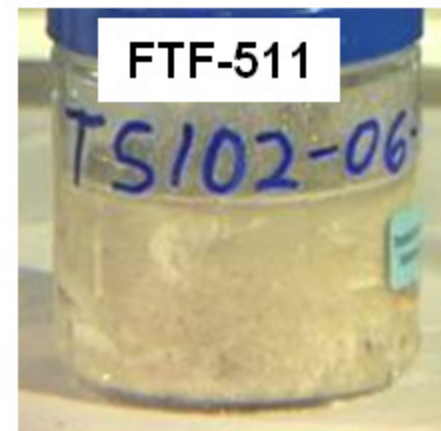
- Ten samples total (seven plus three)
- Top three samples, wet with Free Liquid
- Next two samples, soft and moldable
- Next two, progressively harder, drier



# Tank 25F Saltcake Core Sample Profile



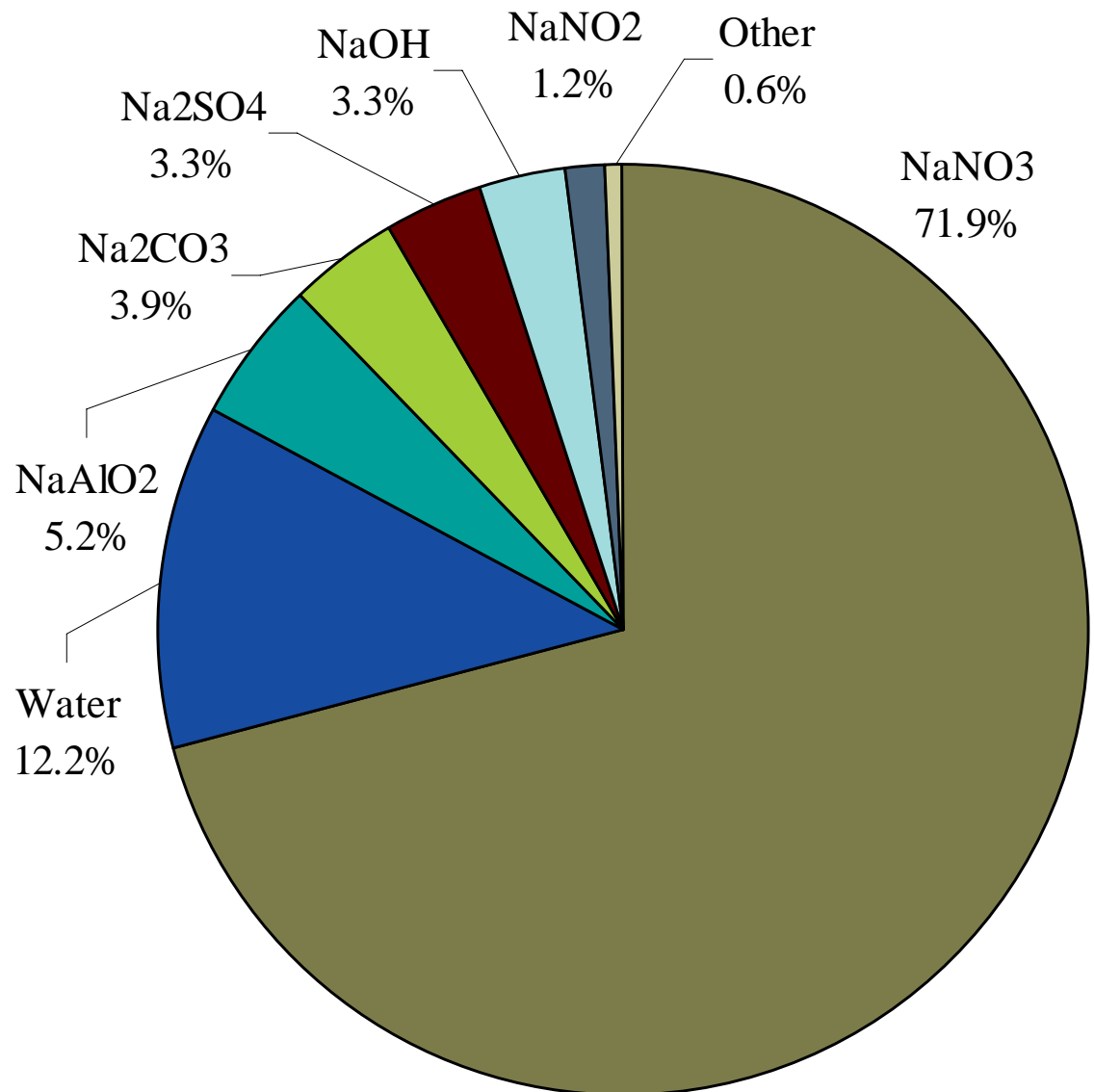
- Ten samples total (seven plus three)
- Top three samples, wet with Free Liquid
- Next two samples, soft and moldable
- Next two, progressively harder, drier
- Bottom three, hard and dry, rewetted



# Tank 25F Composite

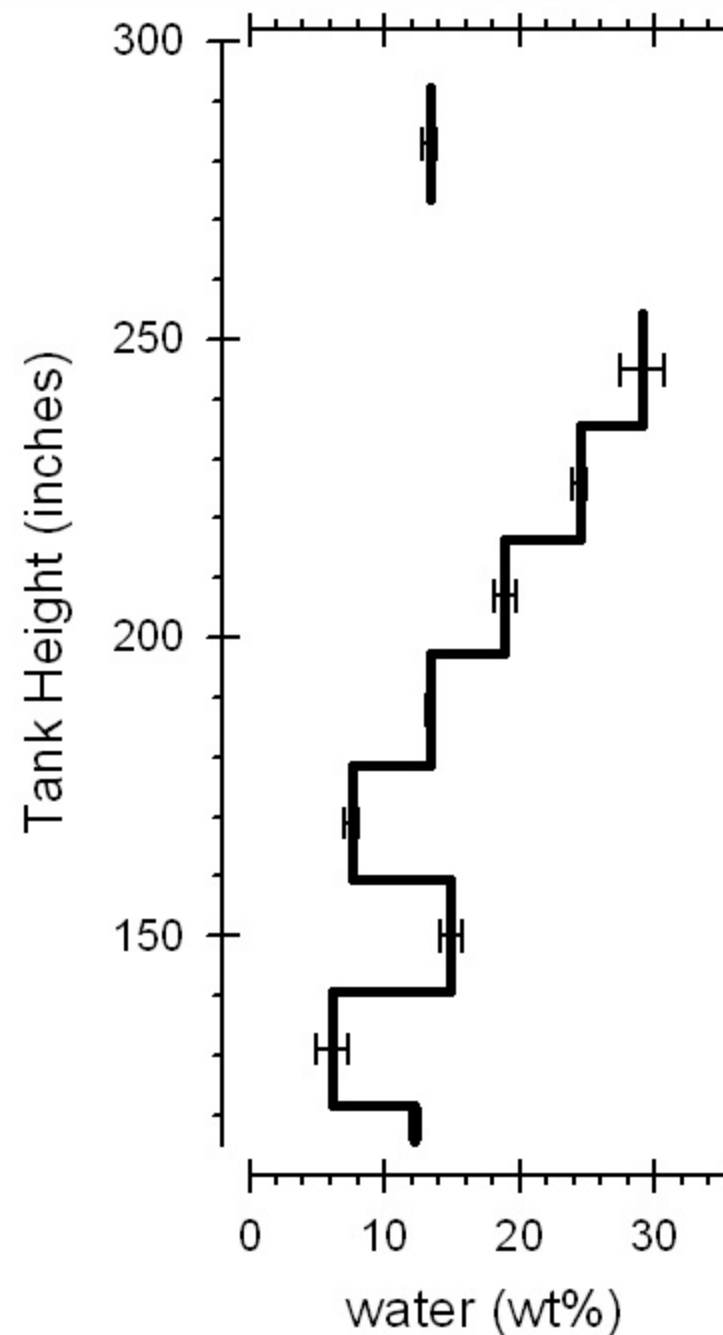
## ■ Undrained Saltcake

- Mostly  $\text{NaNO}_3$
- Significant Al
- Moderate moisture
- SpG = 1.92 (re-packed)
- Cs-137 of 0.68 Ci/gal
- Total Pu alpha of <60 nCi/g



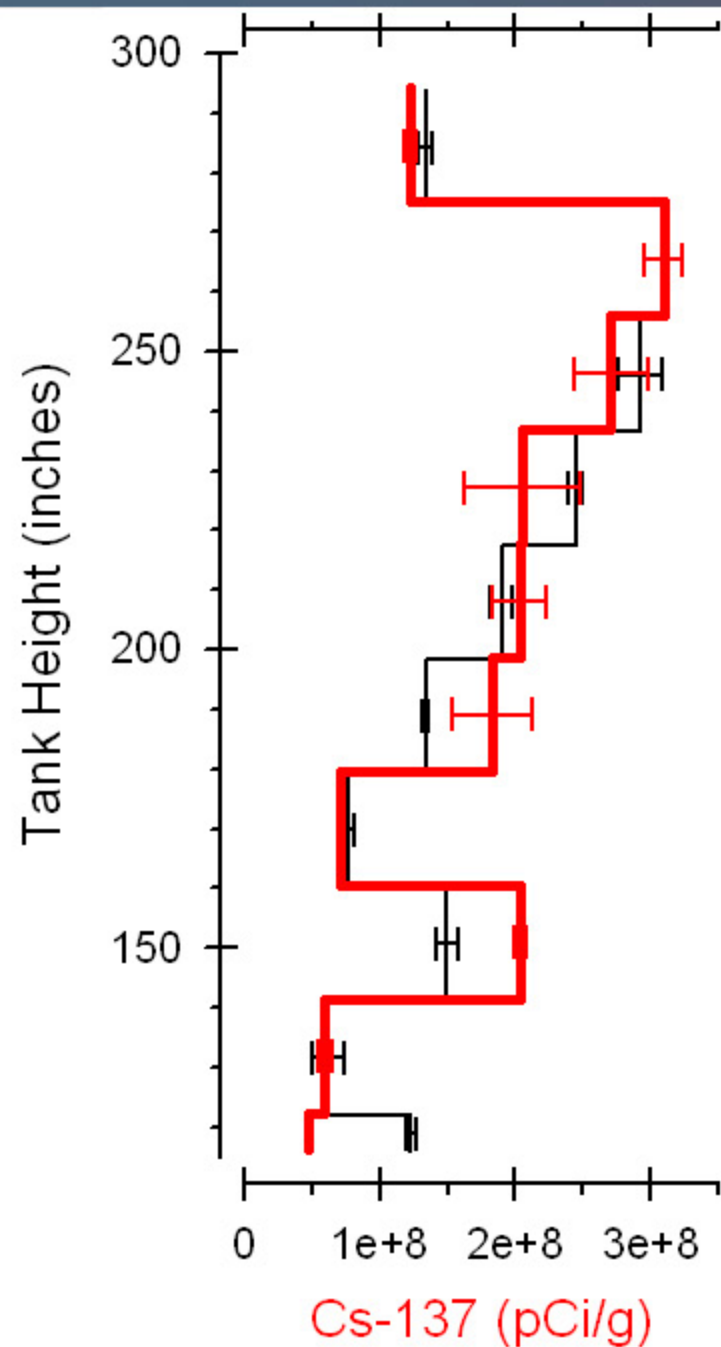


# Tank 25F Segment-to-Segment Profile



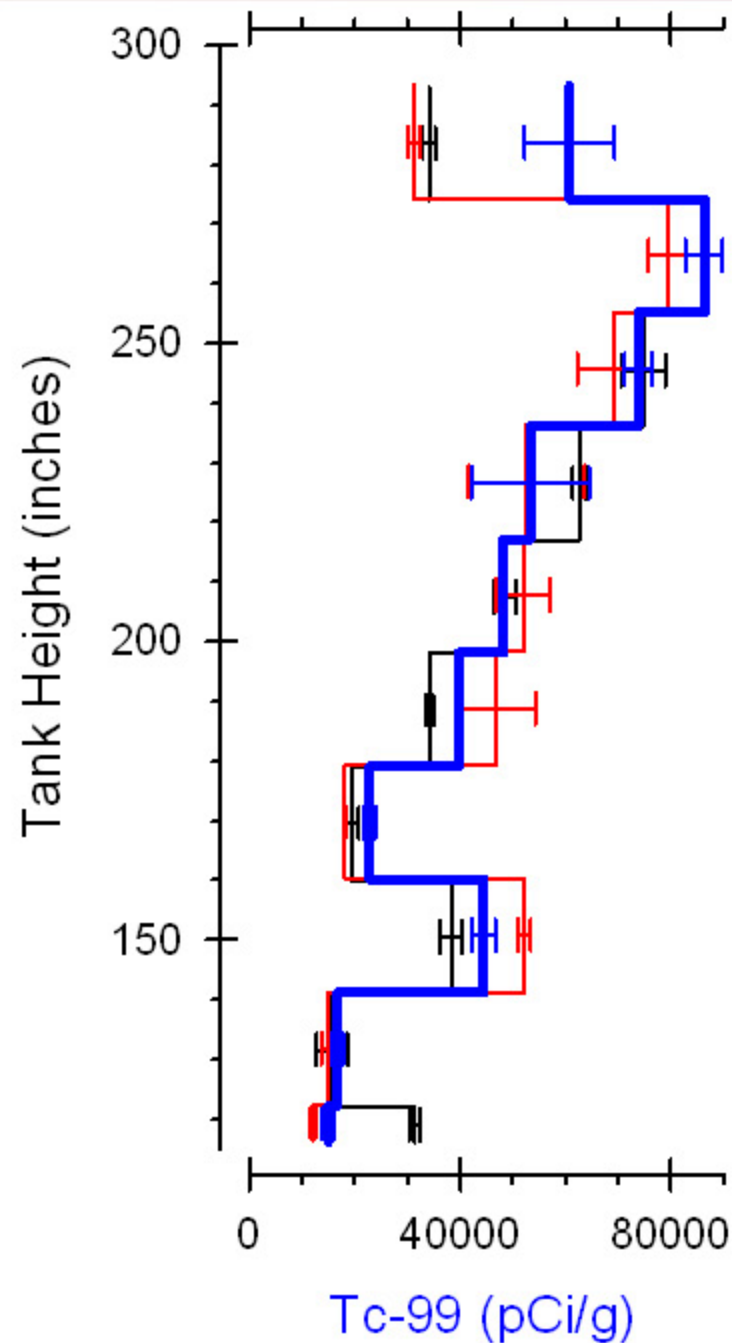
- **Analyte Correlations**
  - Bivariate analysis statistics

# Tank 25F Segment-to-Segment Profile



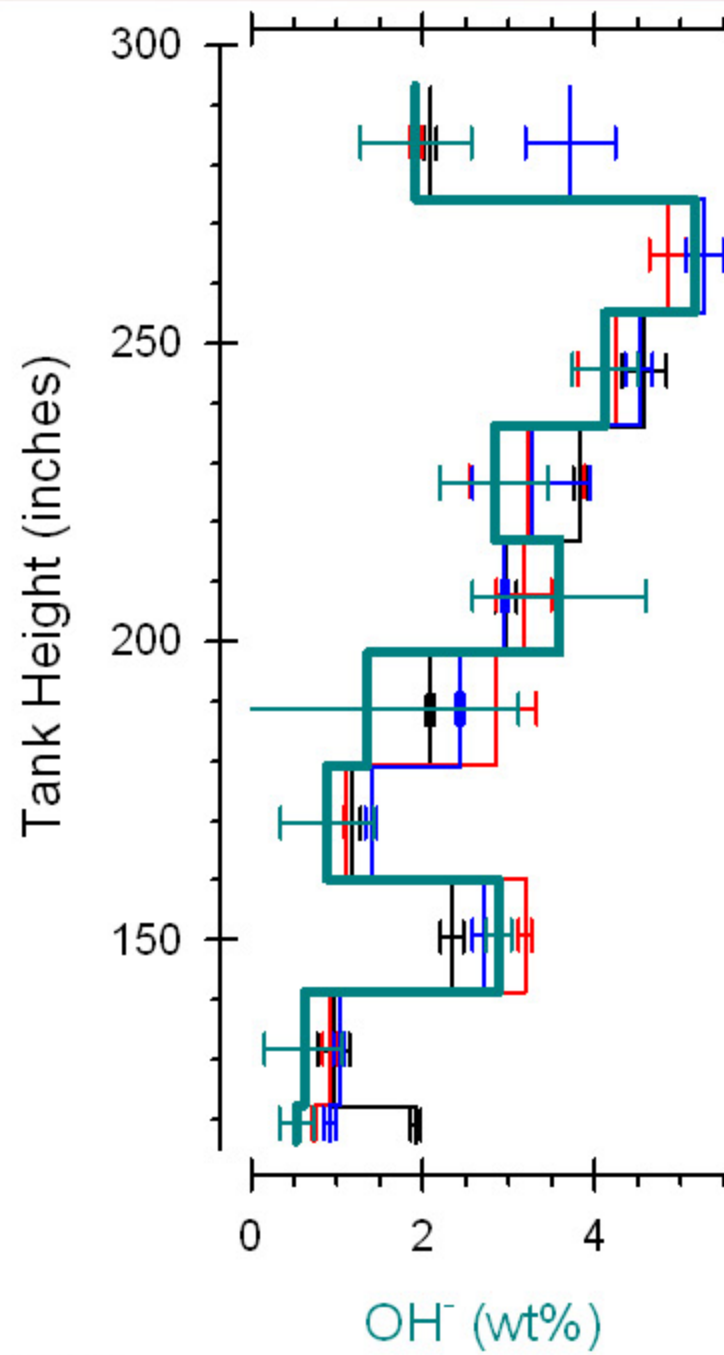
- **Analyte Correlations**
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- **Moisture content correlated with fully soluble components:**  
water, **Cs-137**, **Tc-99**, **OH**, **NO<sub>2</sub>**

# Tank 25F Segment-to-Segment Profile



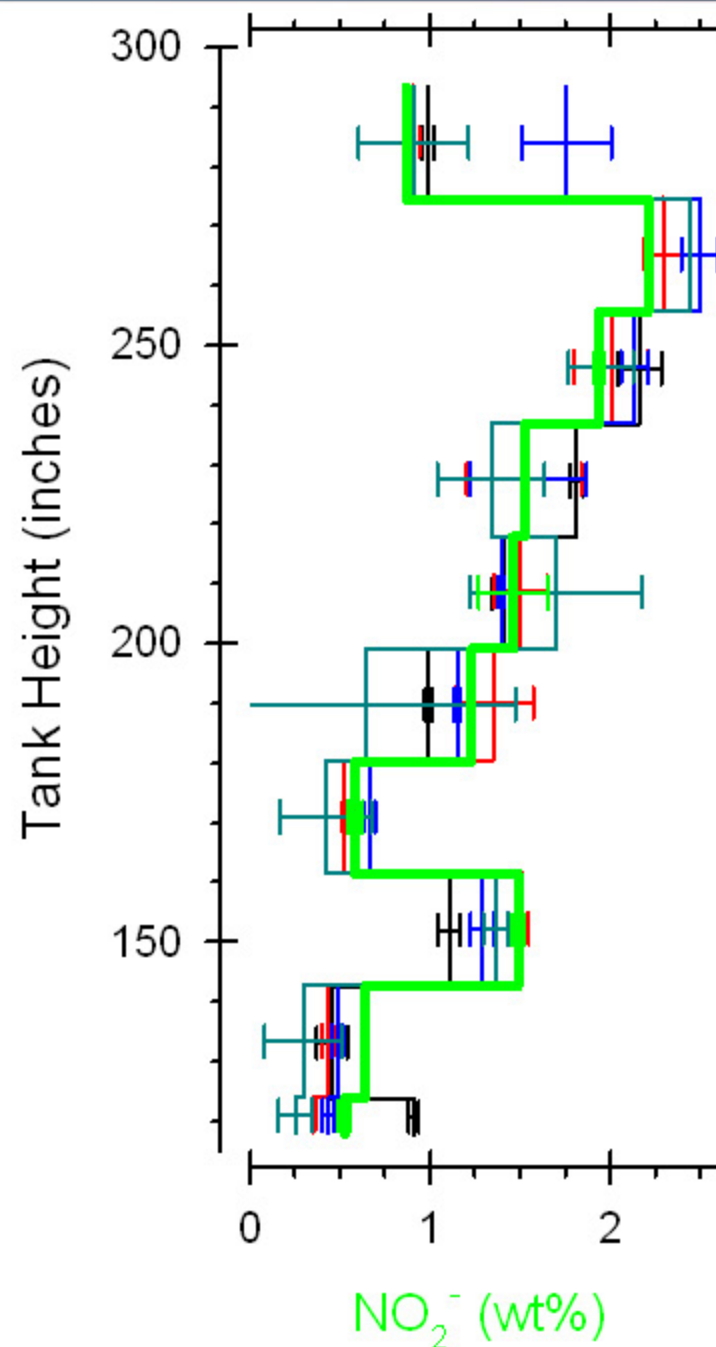
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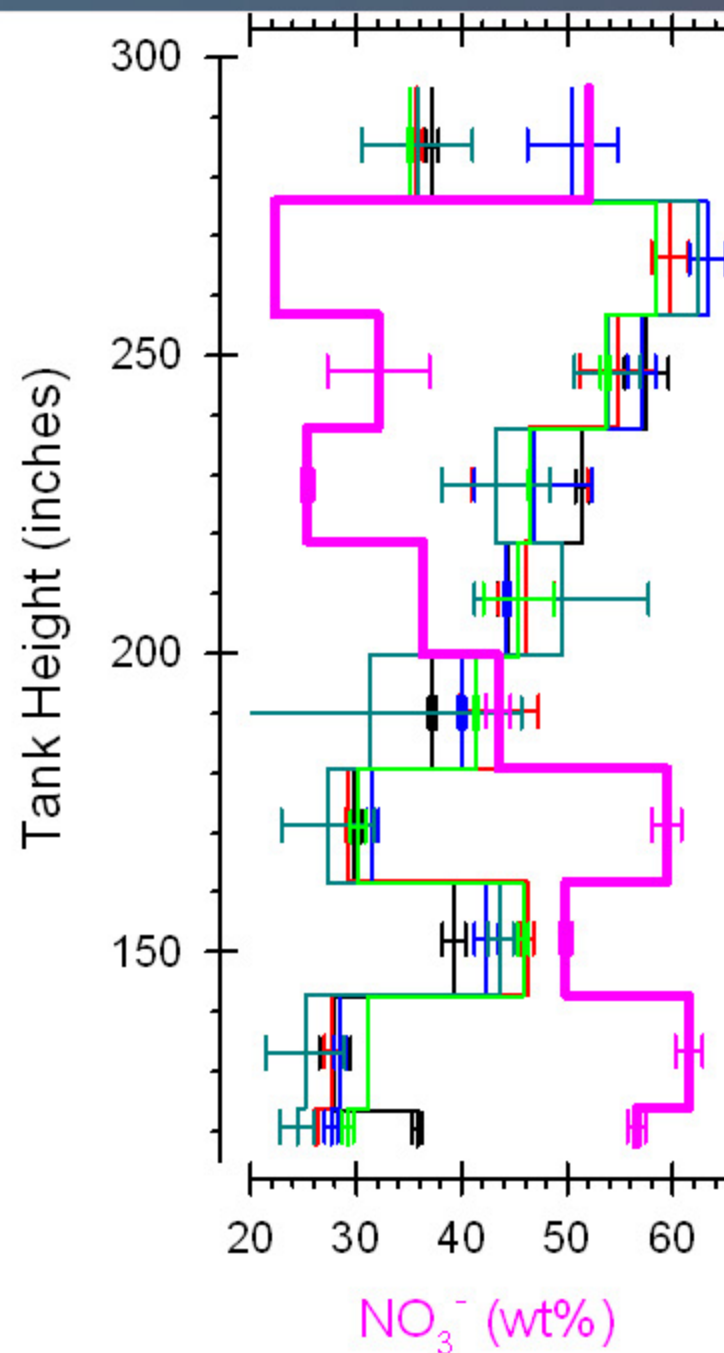
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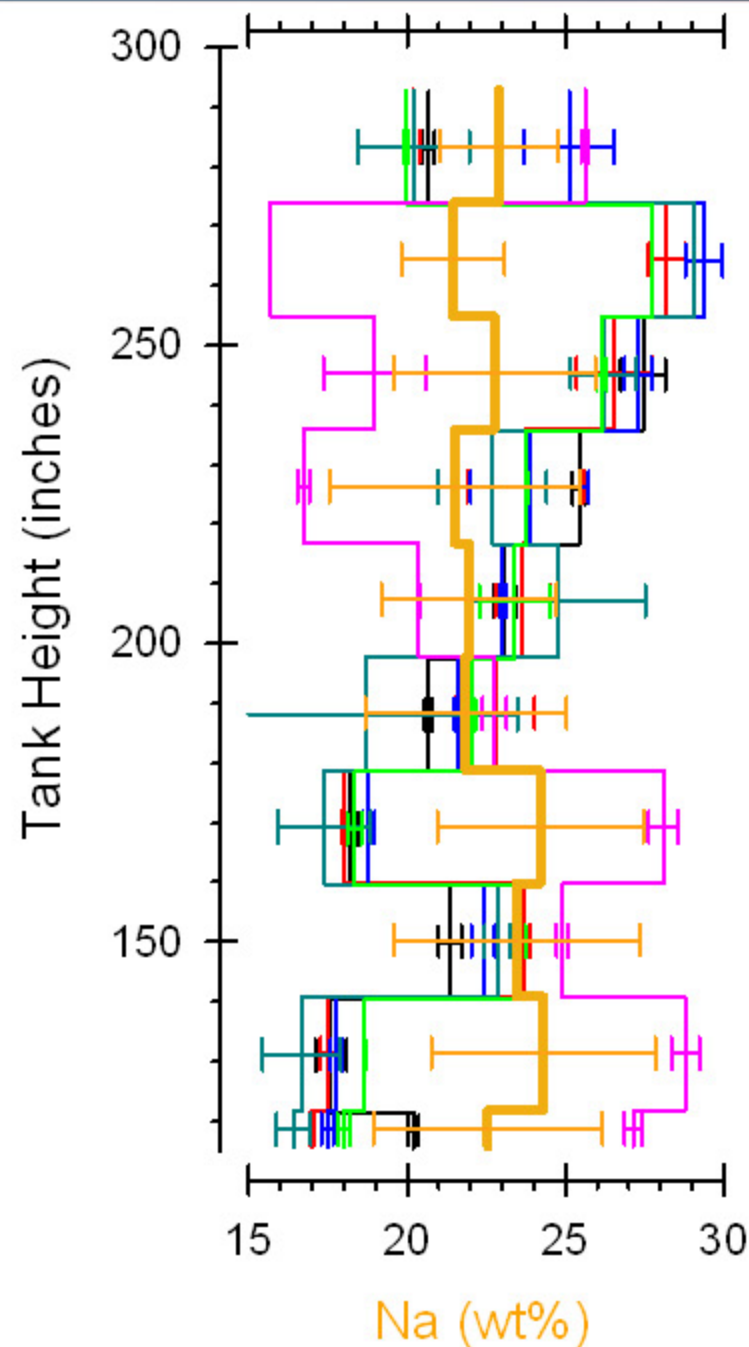


# Tank 25F Segment-to-Segment Profile



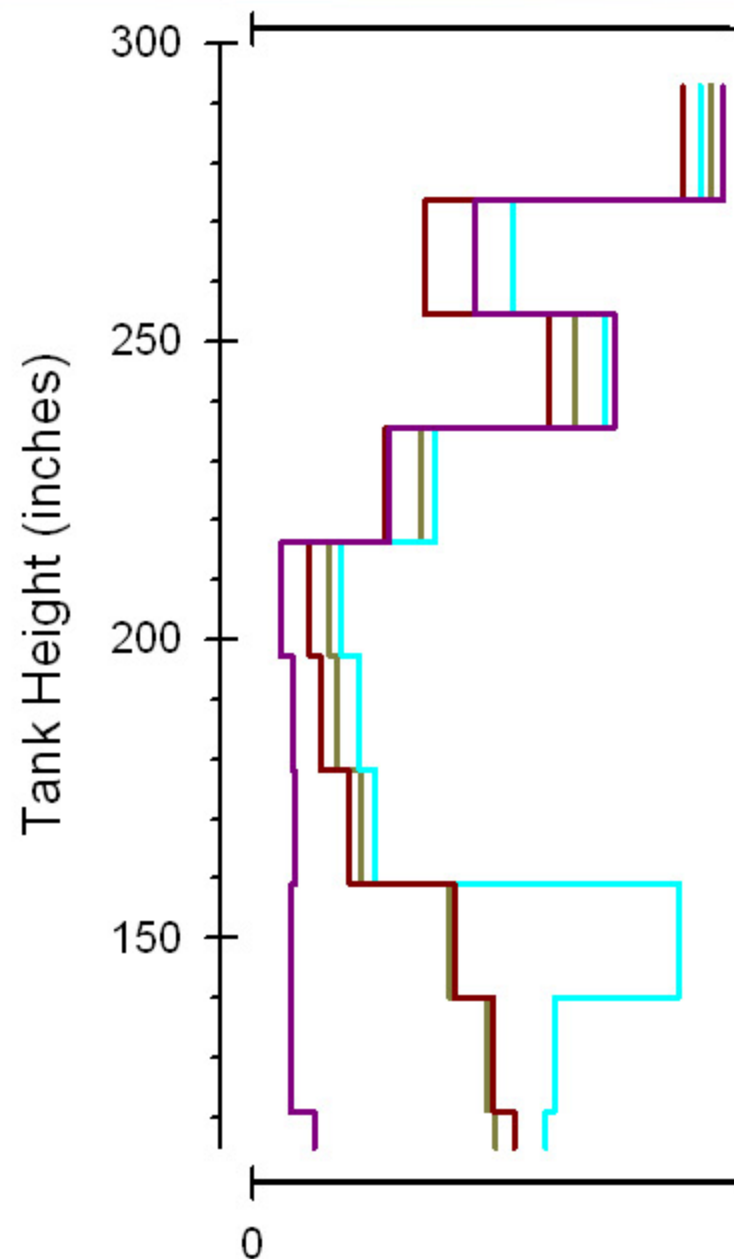
- **Analyte Correlations**
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water,  $\text{Cs-137}$ ,  $\text{Tc-99}$ ,  $\text{OH}$ ,  $\text{NO}_2$
- **Inverse correlation:**  $\text{NO}_3^-$ ,  $\text{Na}$

# Tank 25F Segment-to-Segment Profile



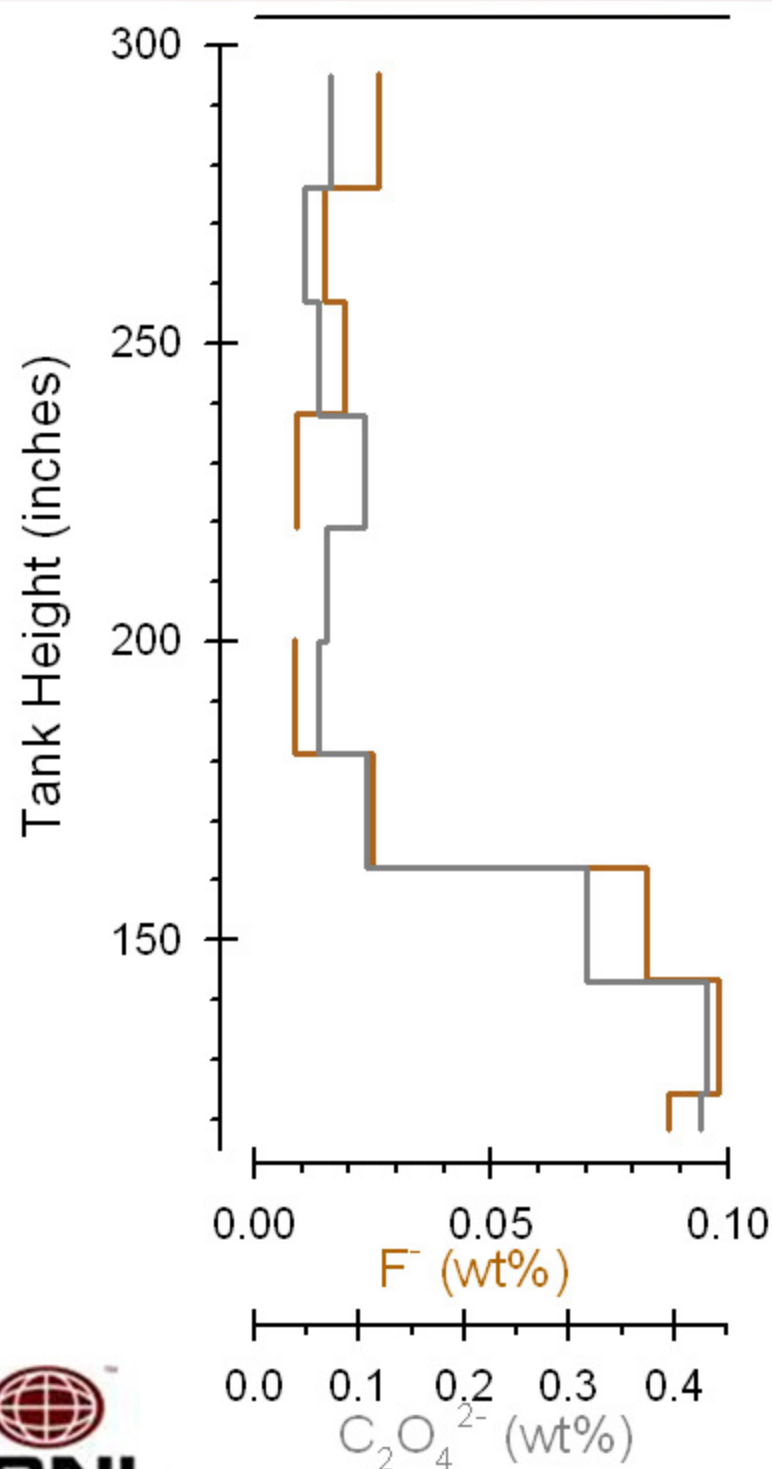
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# Tank 25F Segment-to-Segment Profile



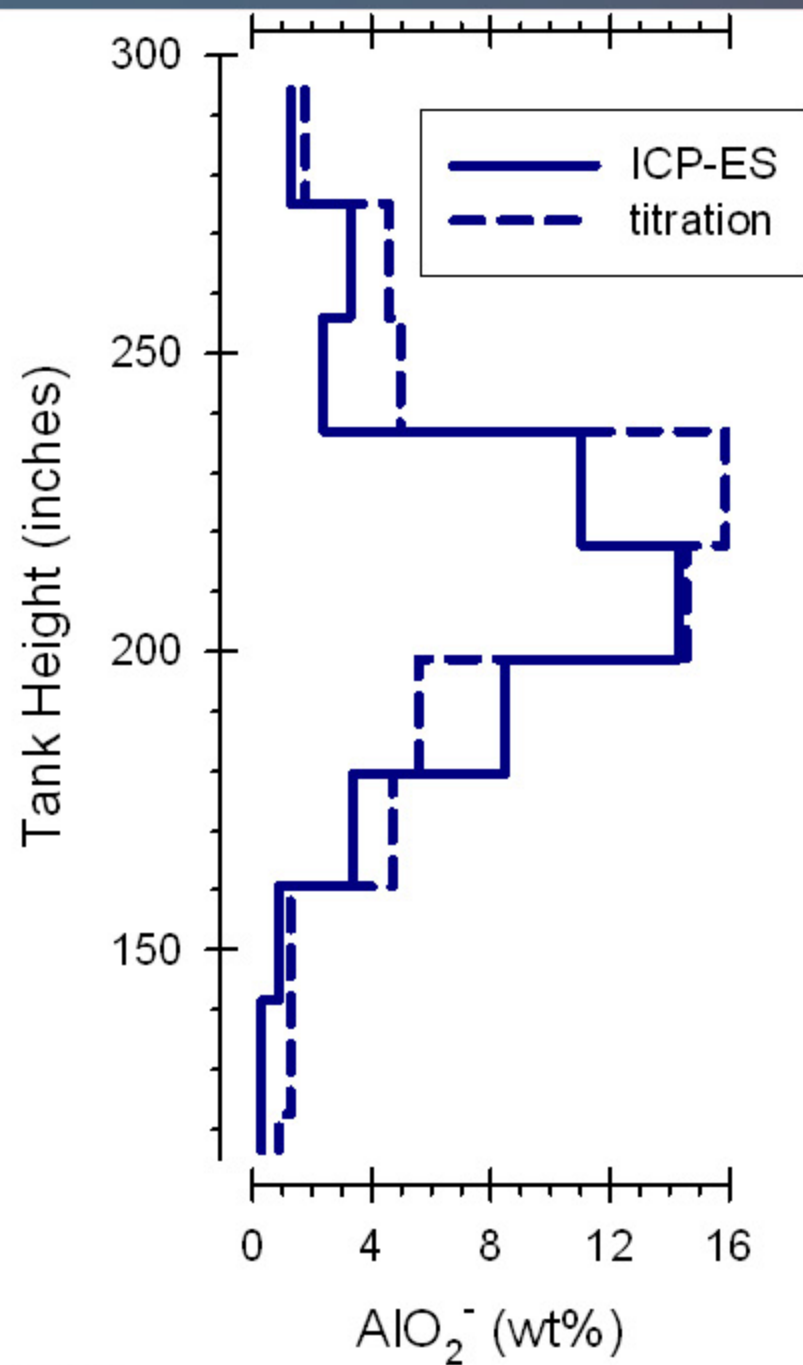
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- **Sludge components:**  
**Pu alpha**, **Sr-90**, **non-Cs beta**, **U**

# Tank 25F Segment-to-Segment Profile



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- **Miscellaneous correlations:**
  - Oxalate and **fluoride** lower in tank

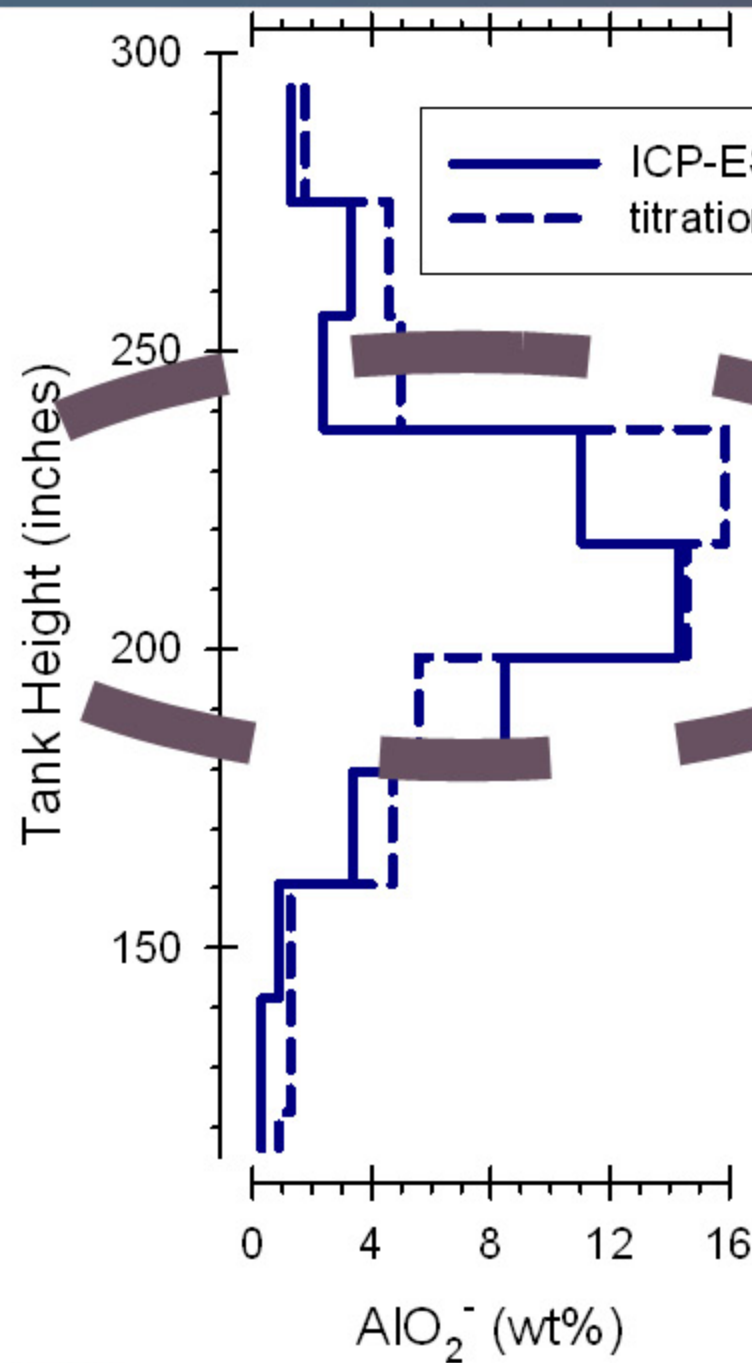
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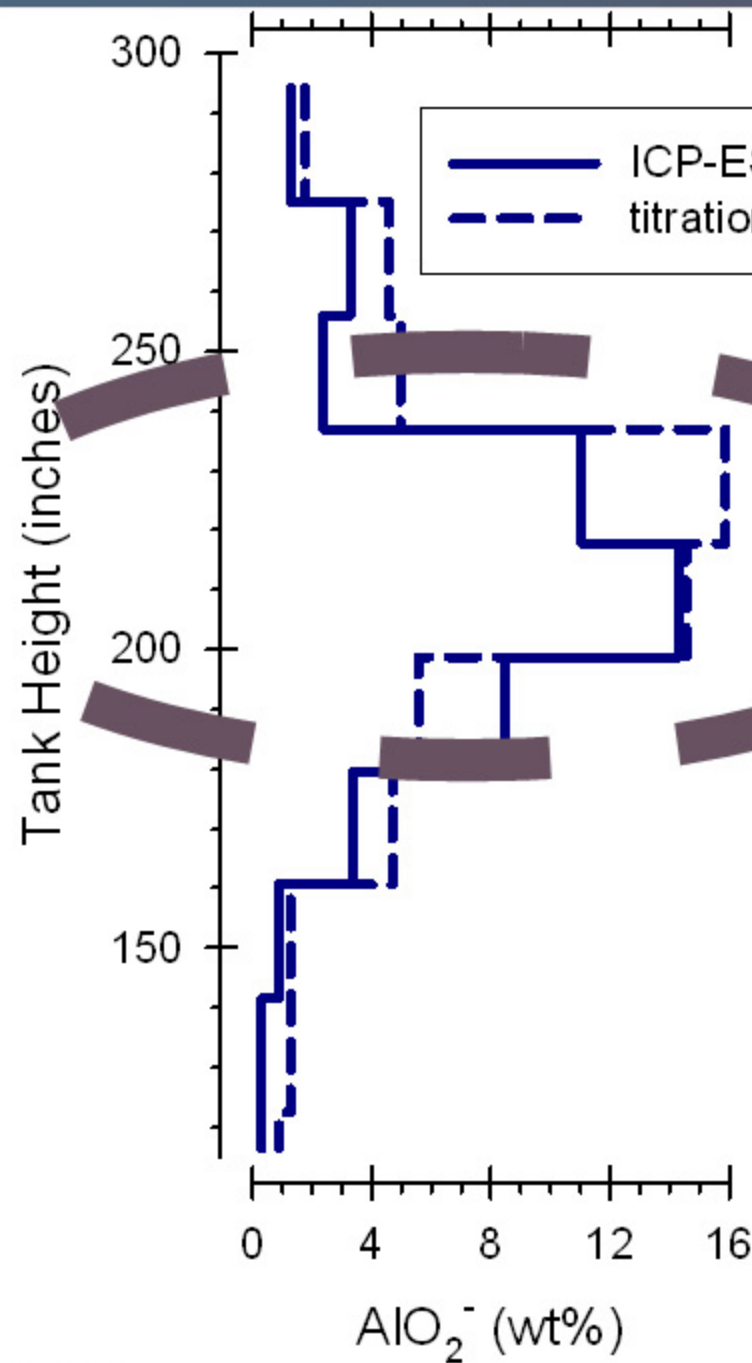


# Tank 25F Segment-to-Segment Profile



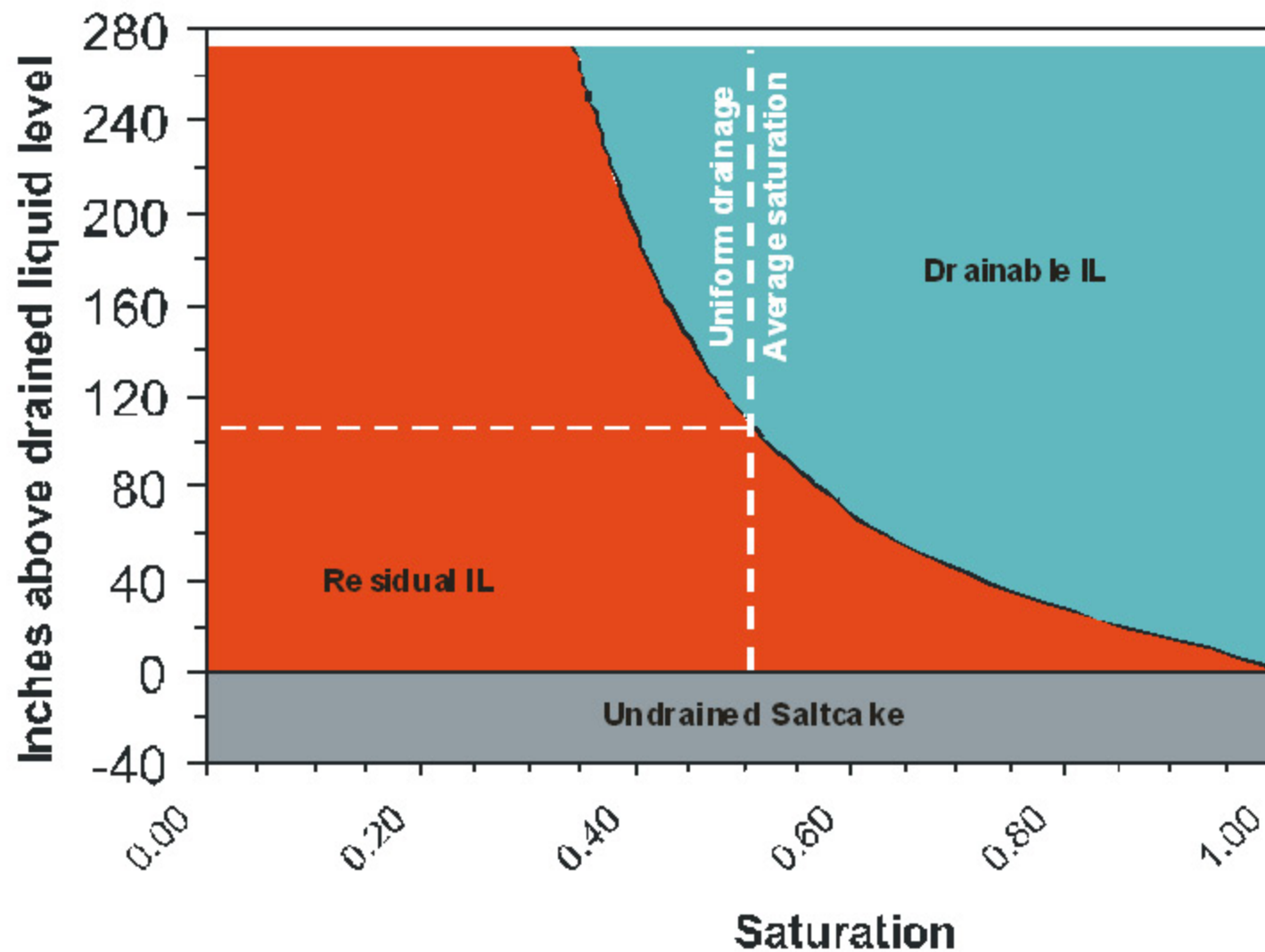
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- **Miscellaneous correlations:**
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- **High **aluminum** in “soft” saltcake**

# Tank 25F Segment-to-Segment Profile



- **Analyte Correlations**
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water, **Cs-137**, **Tc-99**, **OH**, **NO<sub>2</sub>**
- **Inverse correlation:** **NO<sub>3</sub>**, **Na**
- **Sludge components:**  
**Pu alpha**, **Sr-90**, **non-Cs beta**, **U**
- **Miscellaneous correlations:**
  - Oxalate and **fluoride** lower in tank
- **High **aluminum** in “soft” saltcake**
- **Difficult to justify reduced sampling**

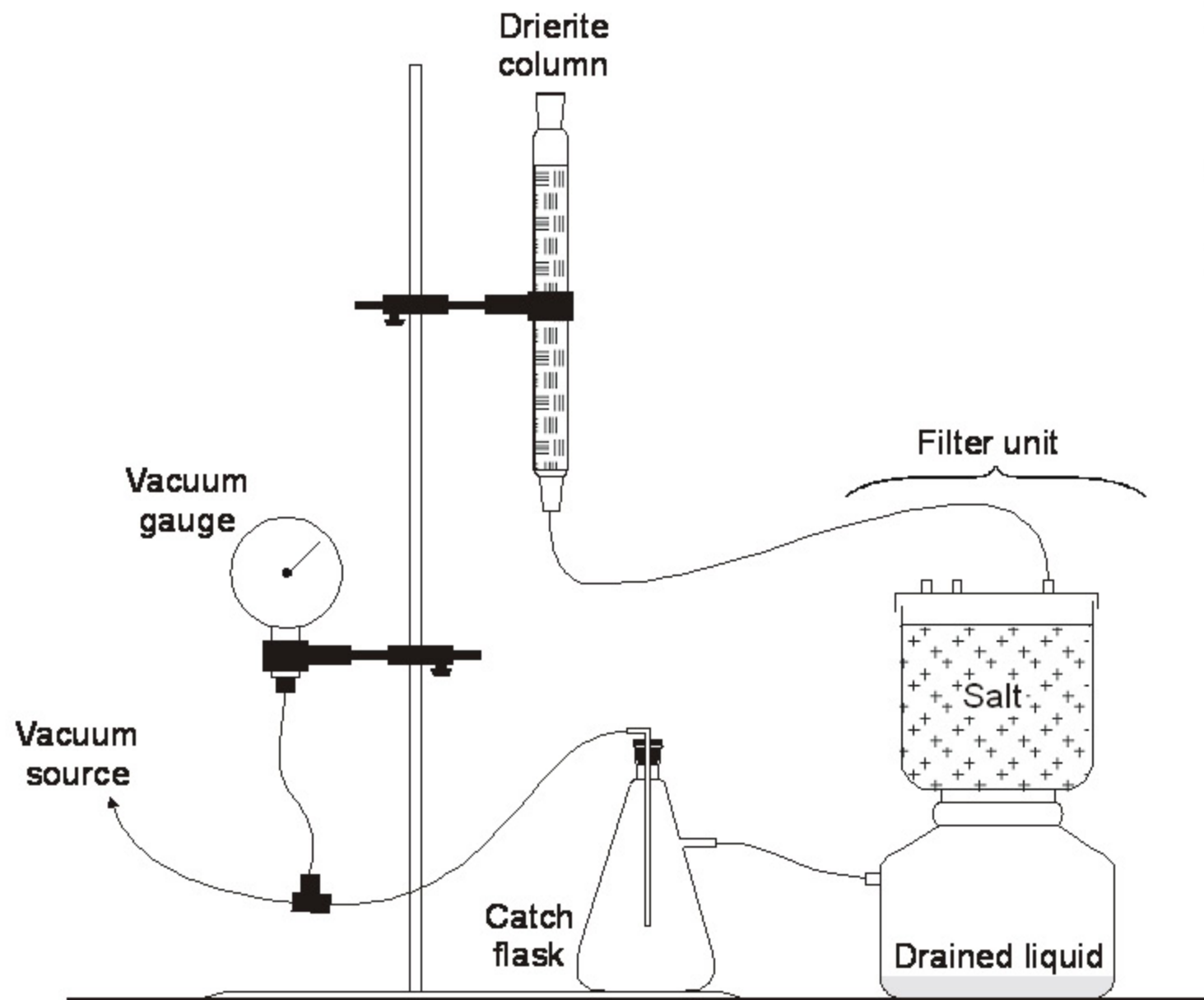
# Tank 25F Saltcake Composite Draining



- Samples obtained prior to draining of Tank 25F
- Draining curve → 11 in. Hg

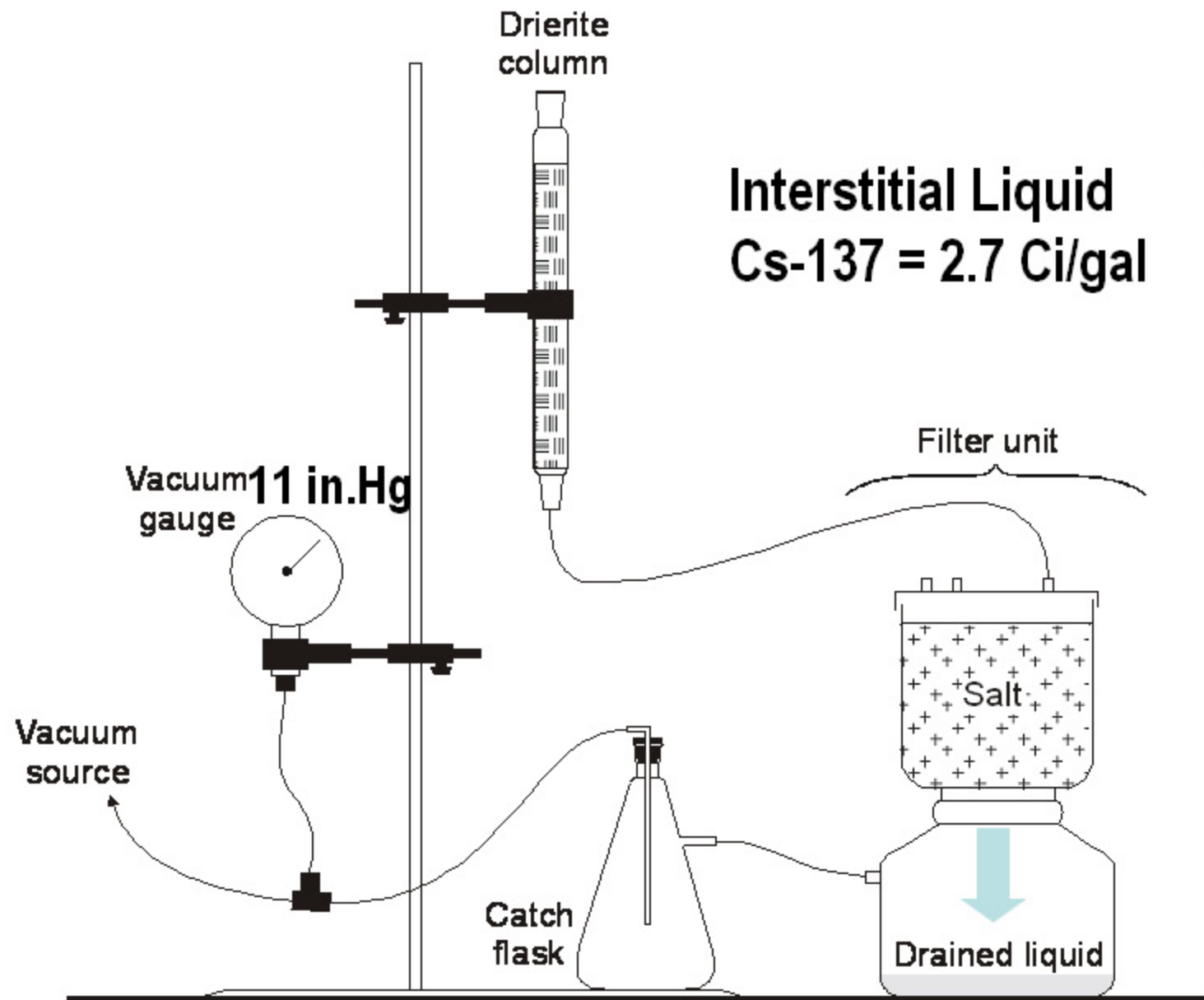


# Tank 25F Saltcake Composite Draining

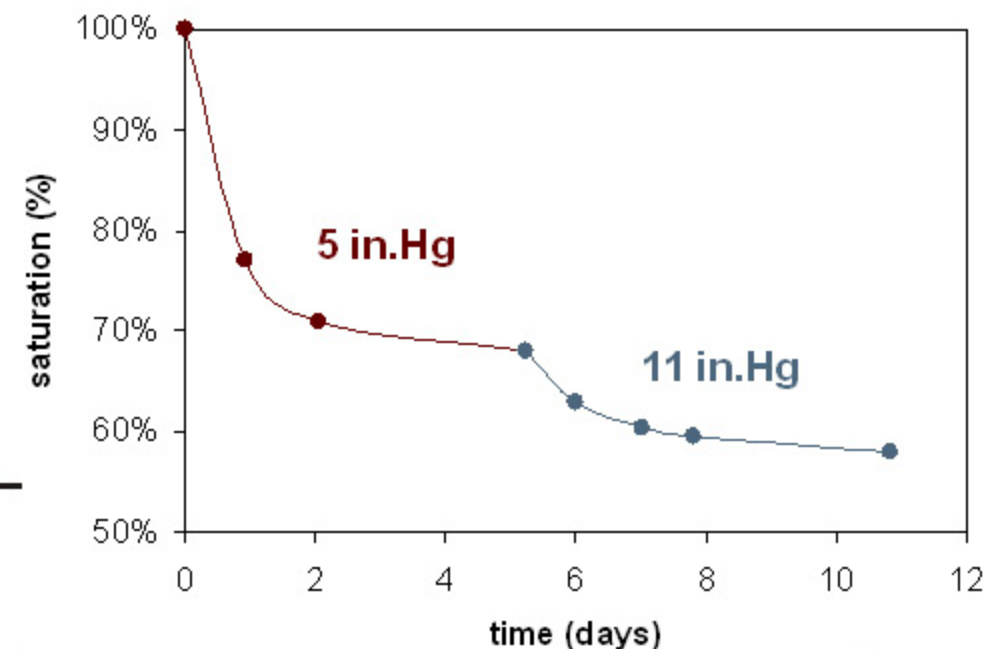


- Samples obtained prior to draining of Tank 25F
- Draining curve  $\rightarrow$  11 in. Hg
- Drained using filter unit

# Tank 25F Saltcake Composite Draining

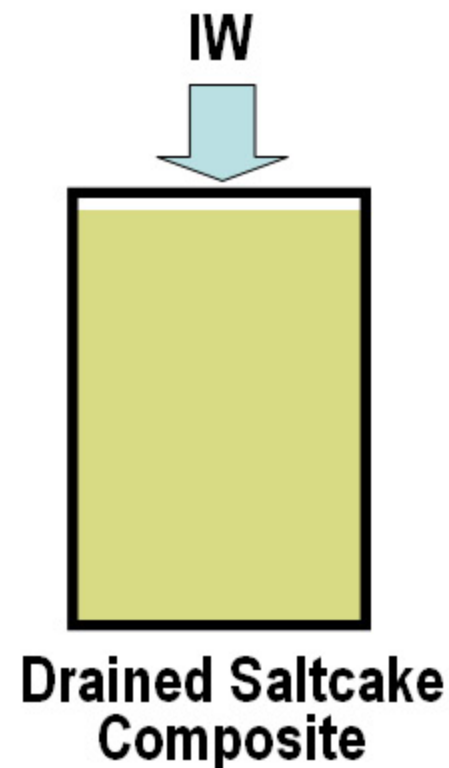


- Samples obtained prior to draining of Tank 25F
- Draining curve  $\rightarrow$  11 in. Hg
- Drained using filter unit
  - 5 days at 5 in. Hg
  - 6 days at 11 in. Hg
  - Subsidence noted
  - Moisture 12.2  $\rightarrow$  7.7 wt%



# Three Batch Dissolution Test

- 650 g of drained saltcake dissolved by 710 g of inhibited water
  - First batch water:salt = 0.25:1 w:w
  - Second batch water:salt = 0.40:1 w:w
  - Third batch water:salt = 0.45:1 w:w
  - Wet residual insoluble solids



# Three Batch Dissolution Test

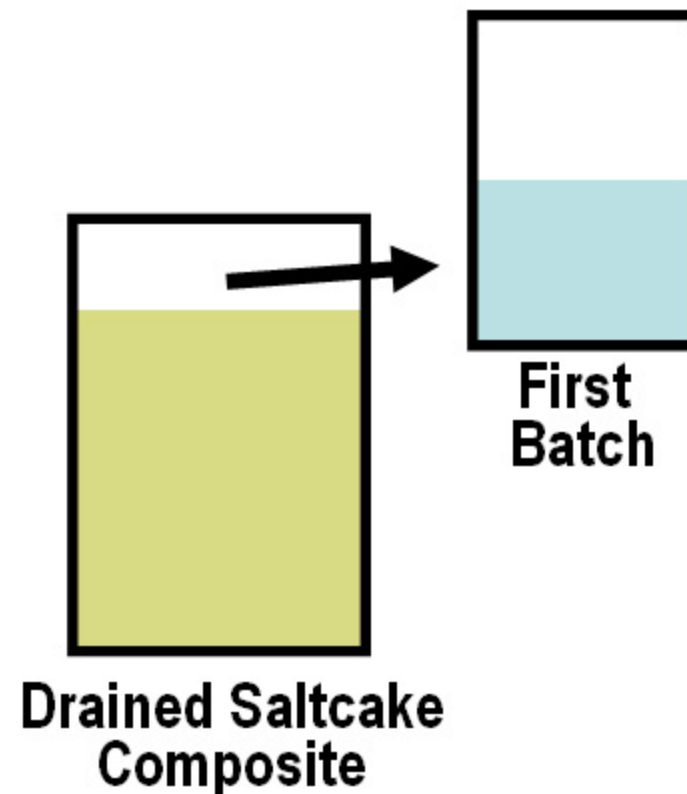
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  - Third batch water:salt = 0.45:1 w:w
  - Wet residual insoluble solids



Drained Saltcake  
Composite

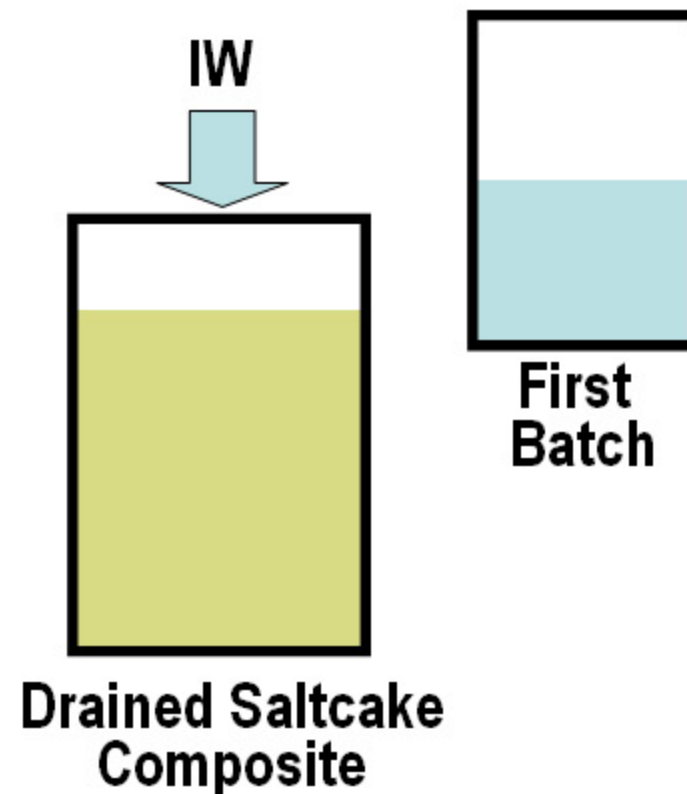
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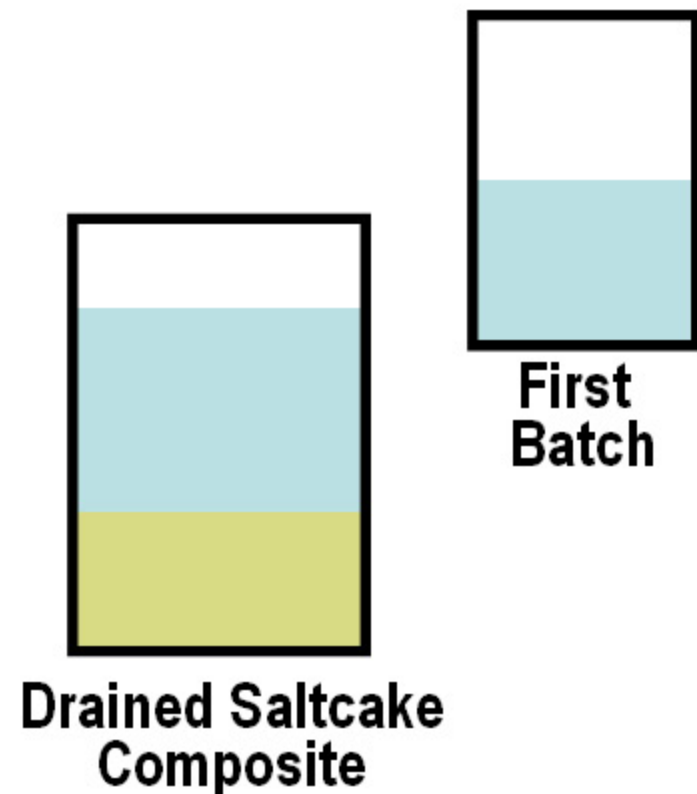
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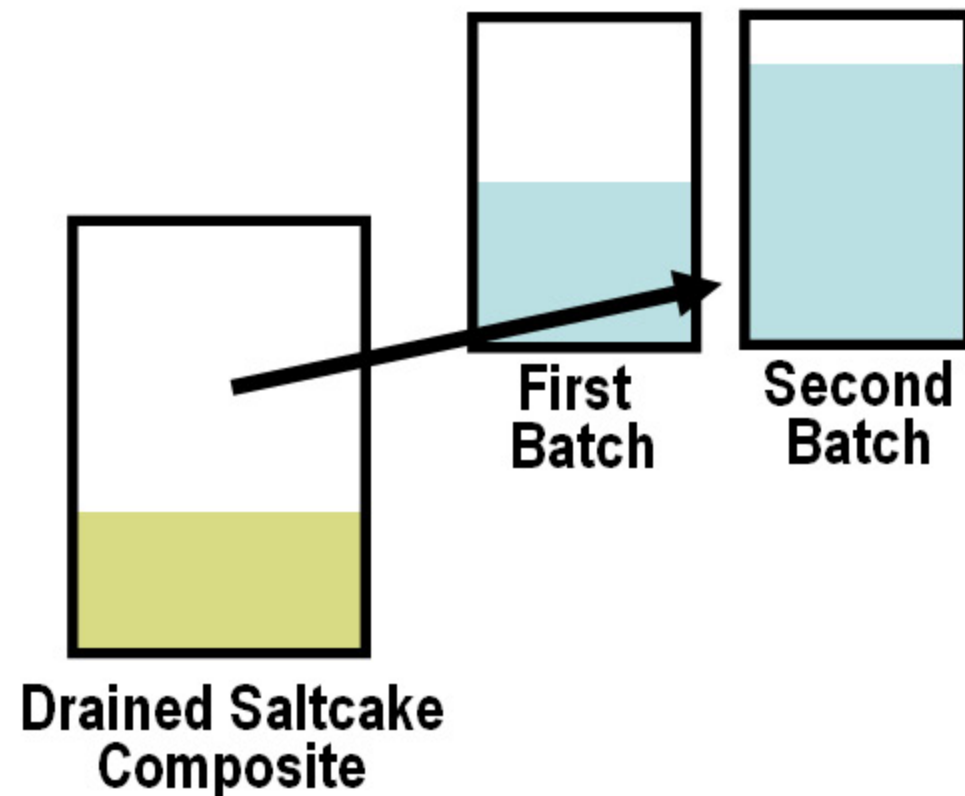
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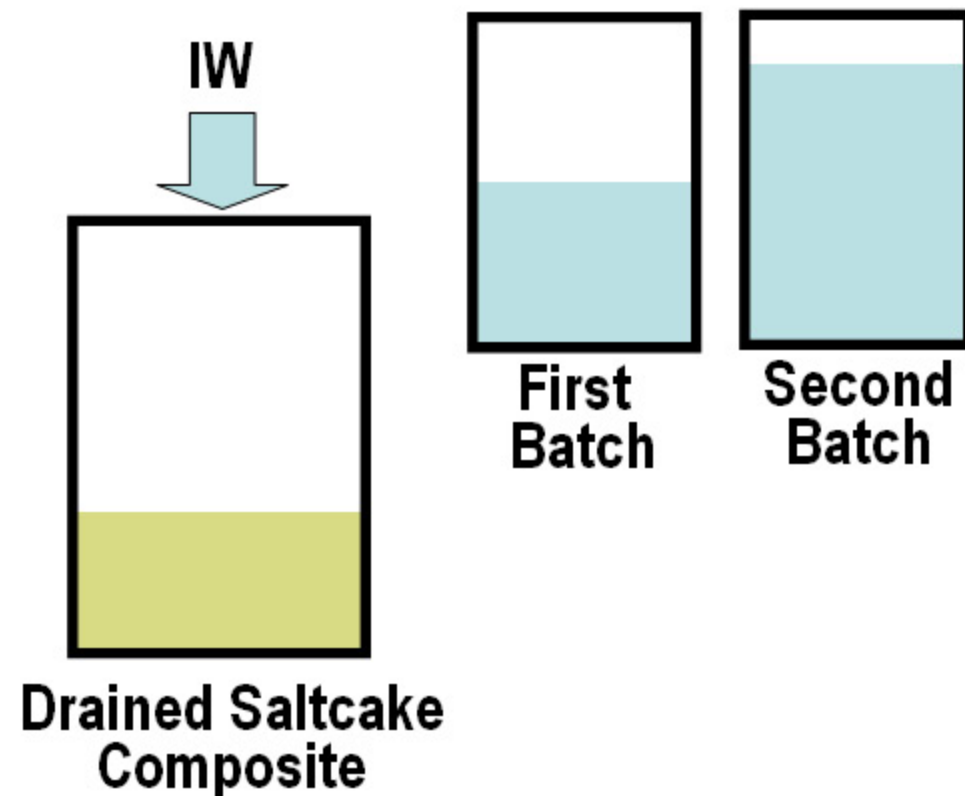
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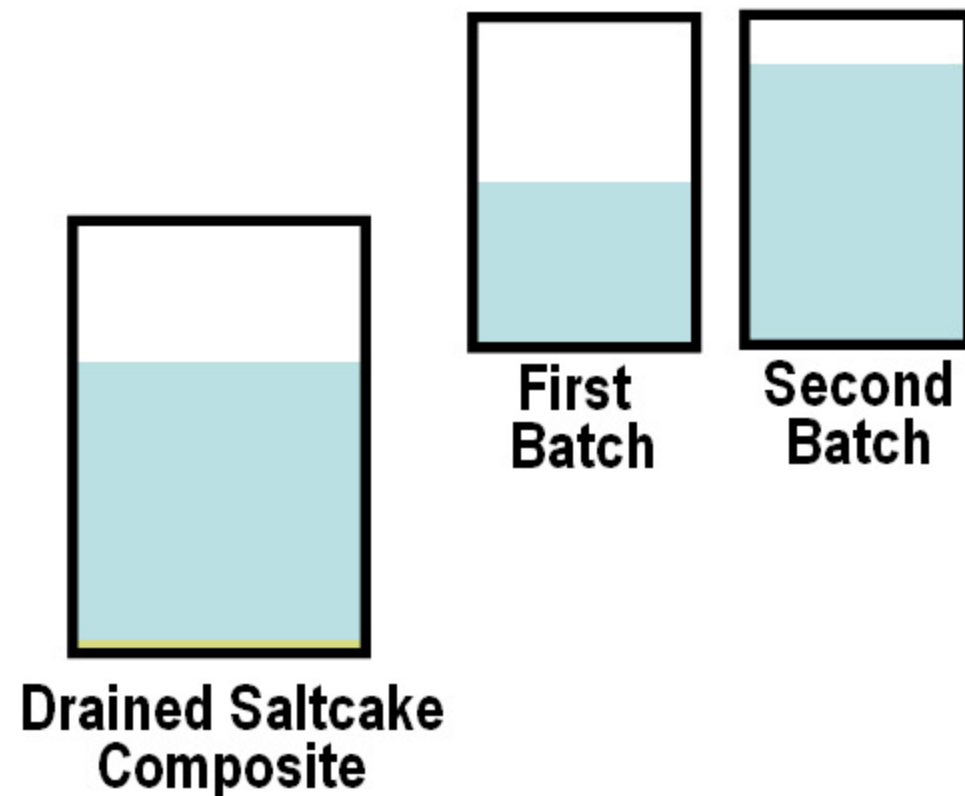
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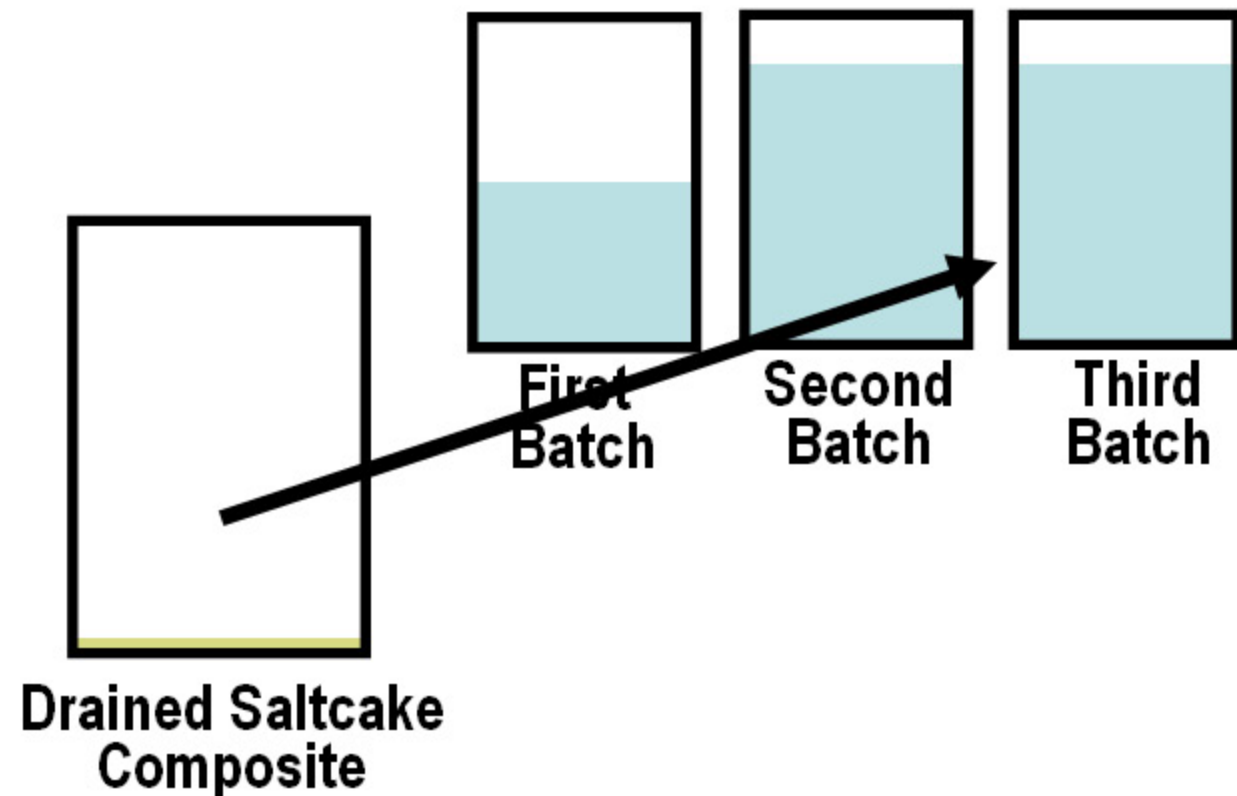
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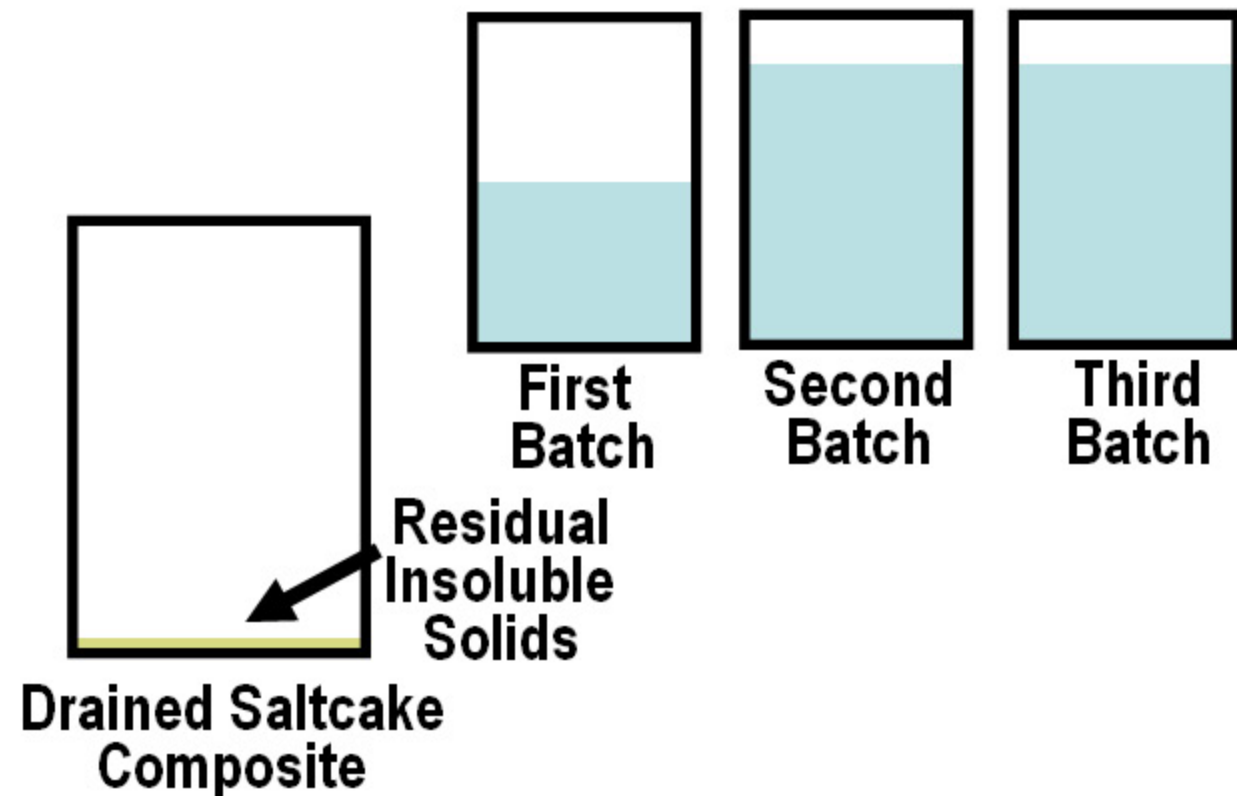
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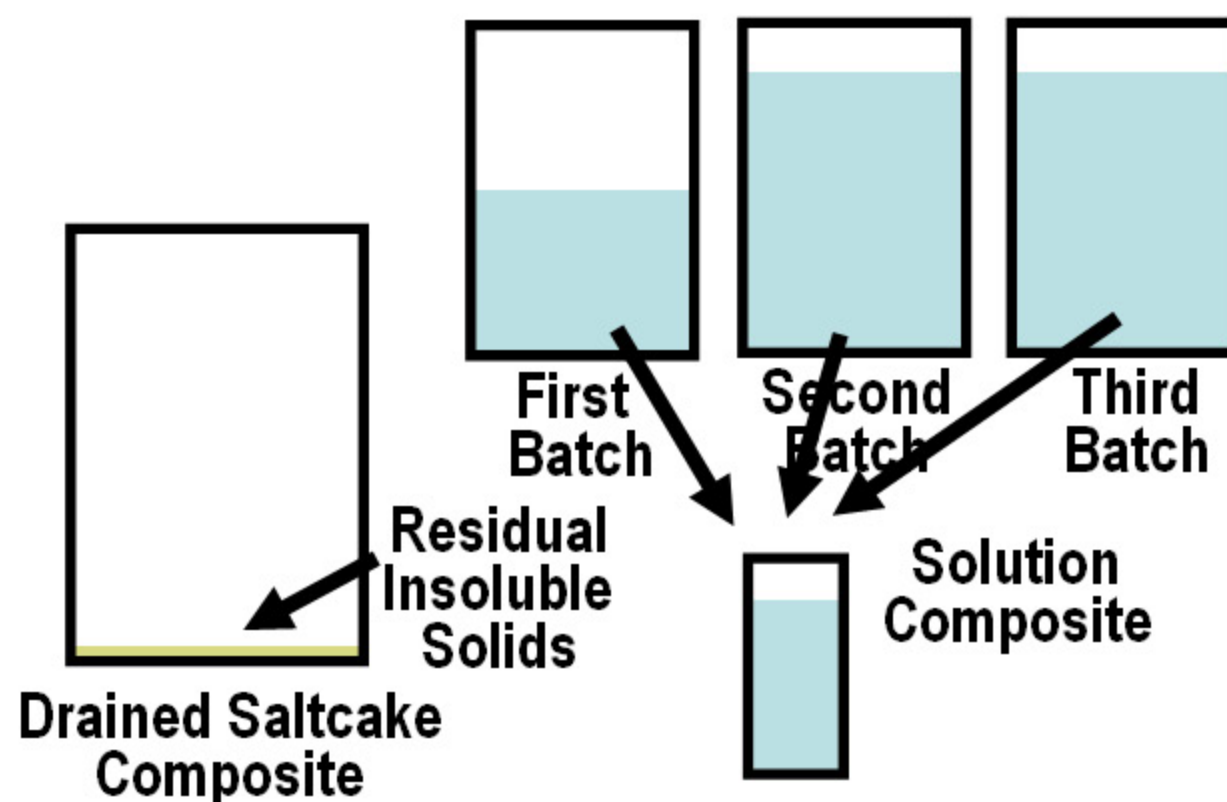
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# Three Batch Dissolution Test

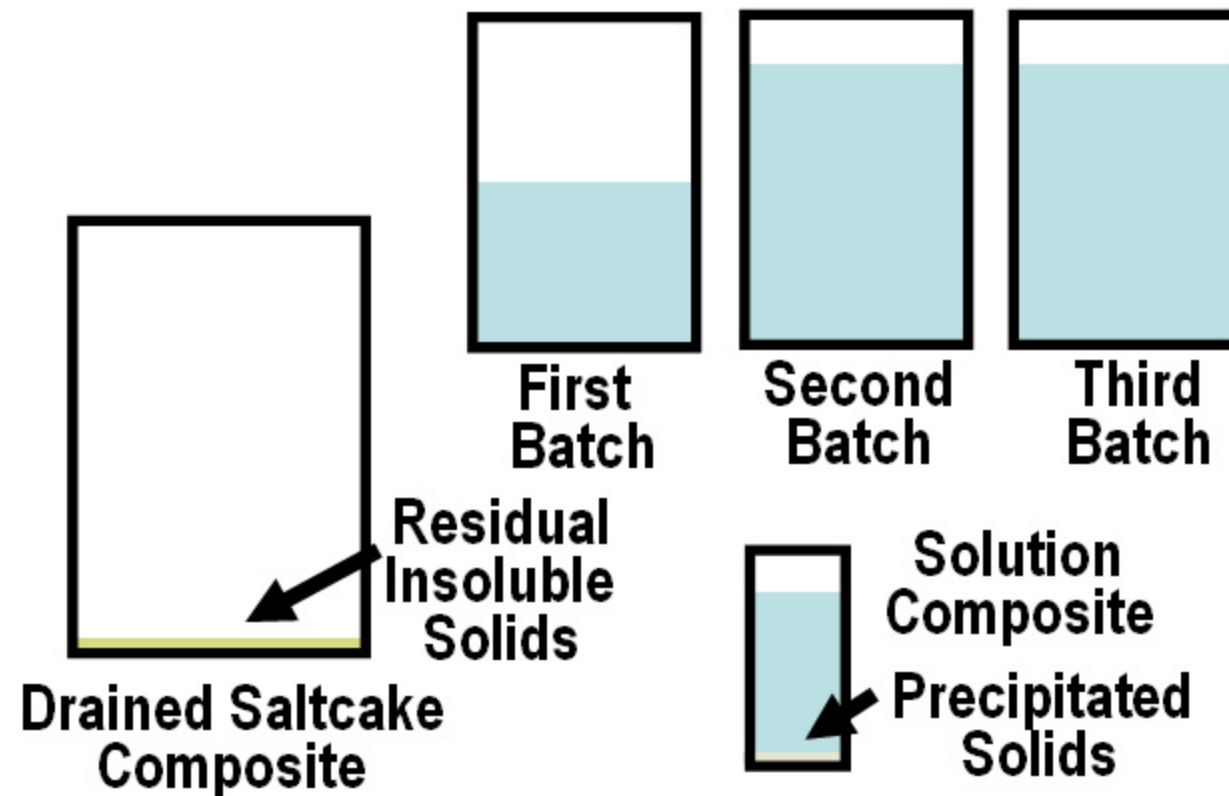
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  - Wet residual insoluble solids
- Solution Composite
  - Used equal percent of each batch
  - Isolated the precipitated solids





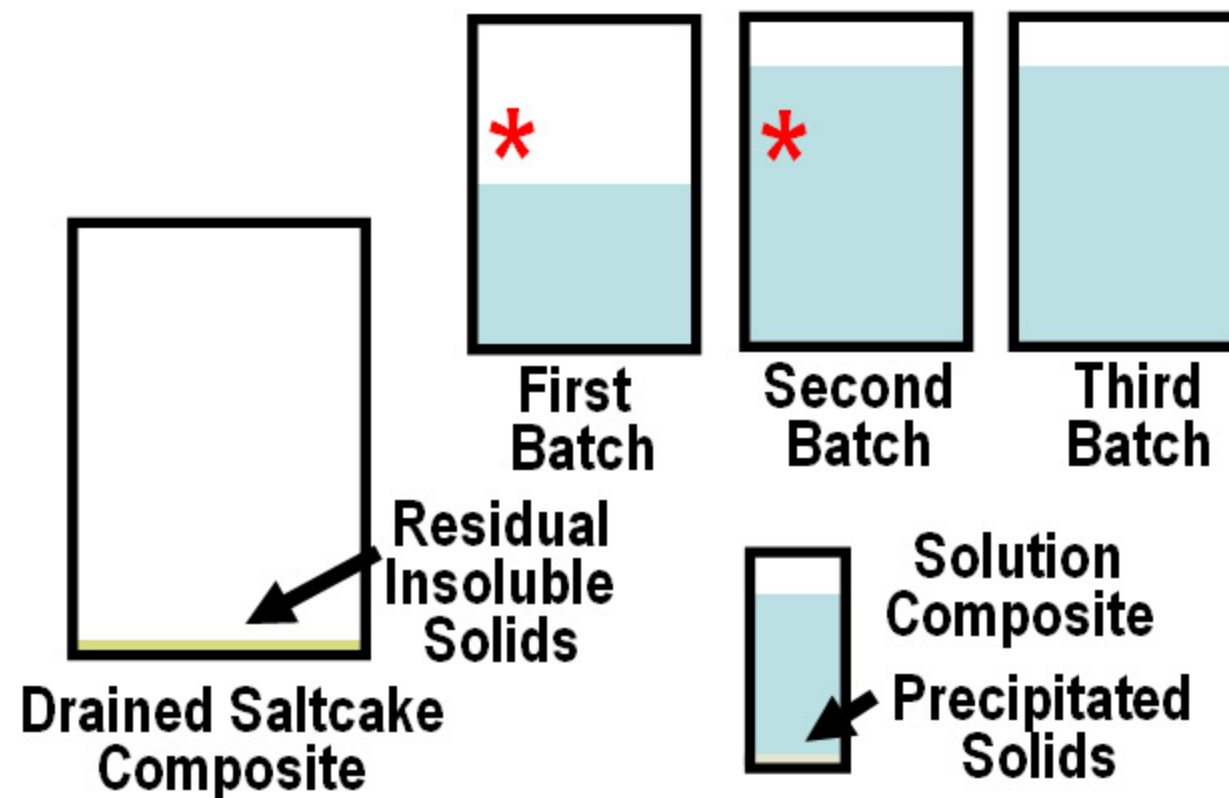
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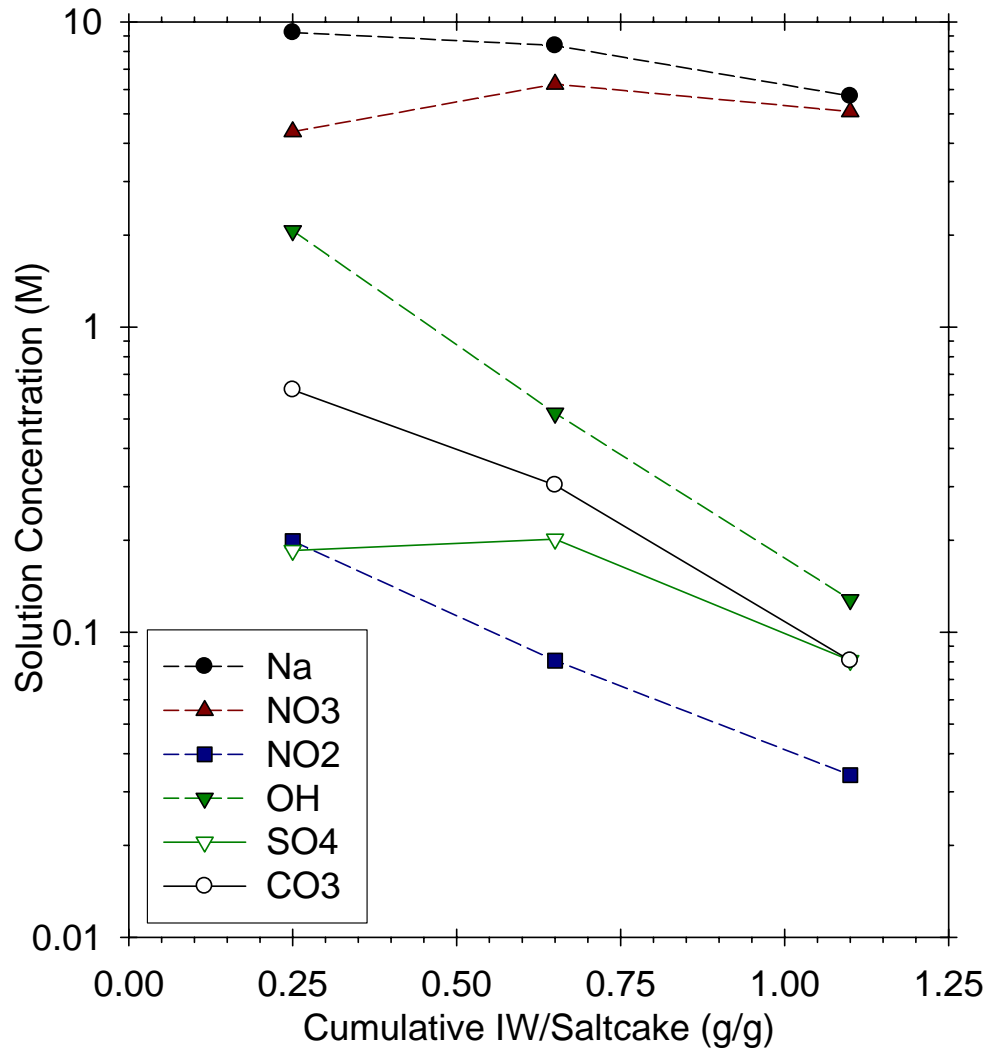


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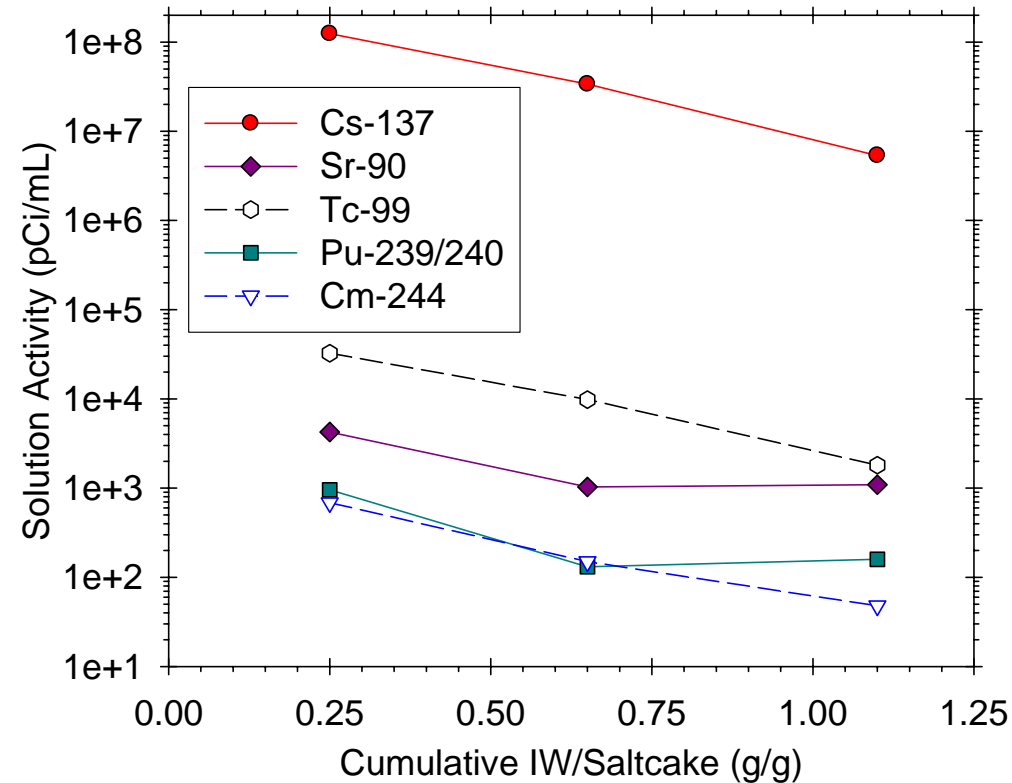
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  - Wet residual insoluble solids
- Solution Composite
  - Used equal percent of each batch
  - Isolated the precipitated solids
  - \* – First and Second batches formed solids over two week period
  - \* – Precipitated solids mostly  $\text{Al}(\text{OH})_3$
  - \* – Al results do not reflect solubility
  - \* – Significant volume (order of 10%)



# Tank 25F Dissolution Profiles

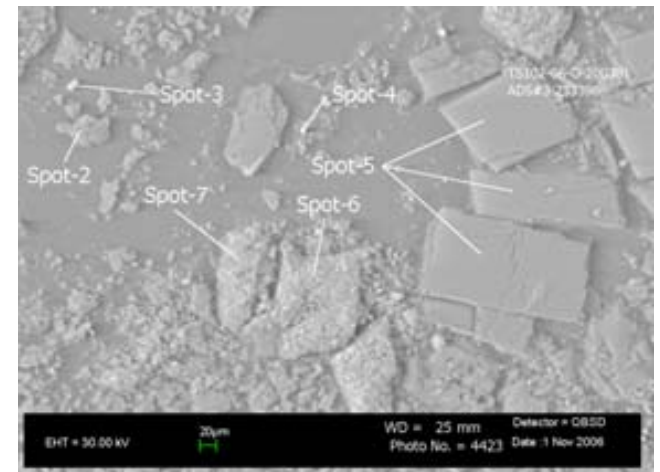
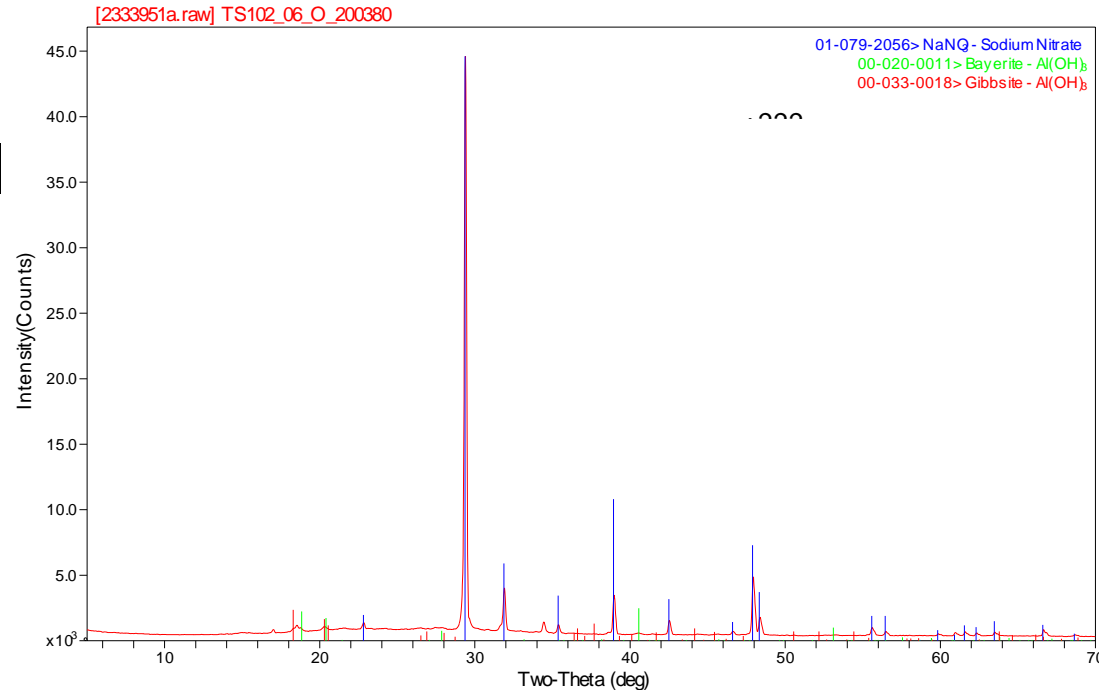


- 93% of salt dissolved
- >50% of solubles in initial 18% soln.
- Na, NO<sub>3</sub>, SO<sub>4</sub> & CO<sub>3</sub> dissolution



# Residual Heel of Solids

- Heel primarily  $\text{NaNO}_3$ 
  - Plus other salts, additional water likely effective
- Metals
  - 2.5 wt% aluminum \*
  - Gibbsite and bayerite
  - 1.1 wt% iron
- Radionuclides
  - >98% of the sample's plutonium and uranium
  - 93% of the non-Cs beta



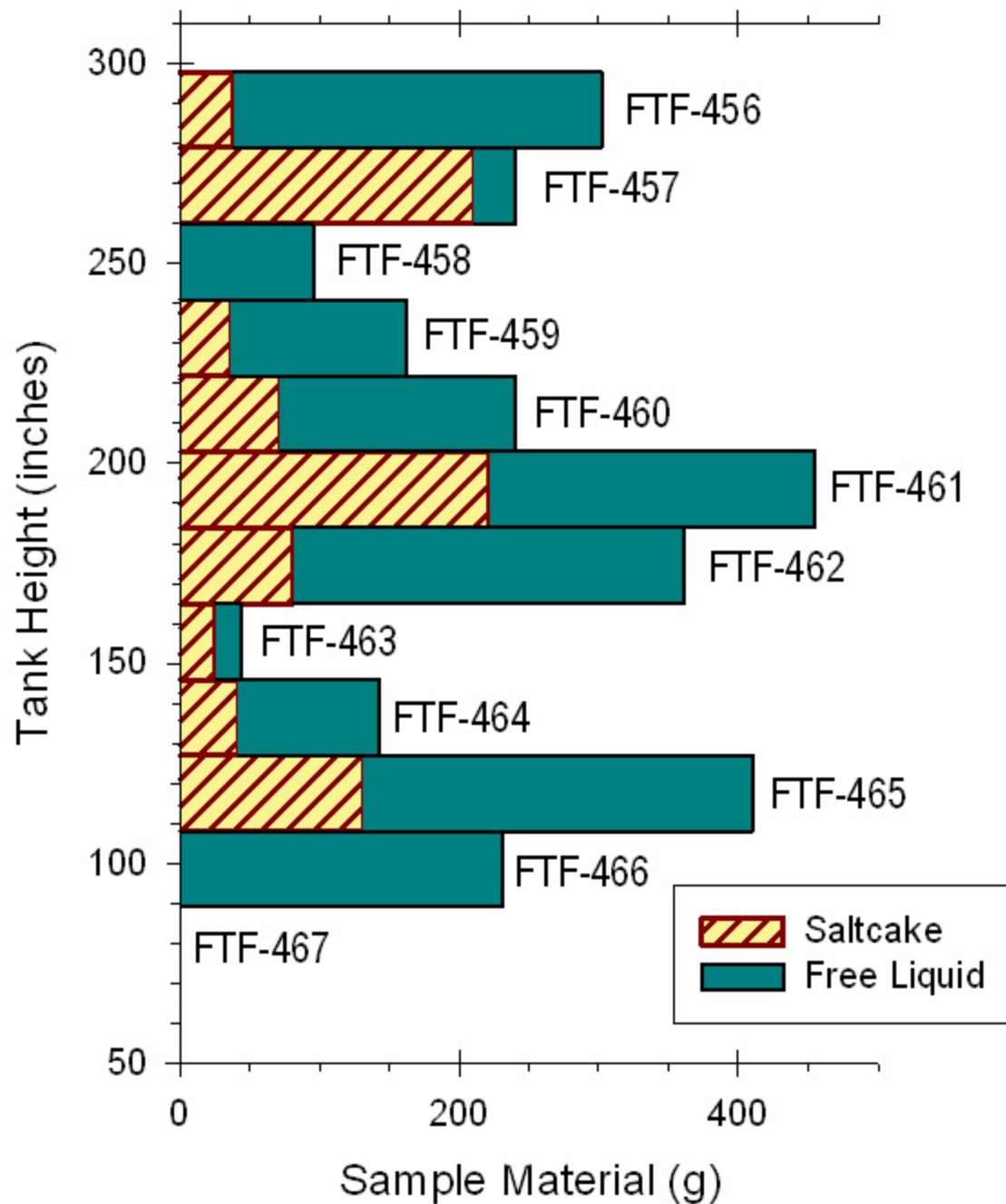
# The Real Benefit of HLW Saltcake Sampling

- Dissolved saltcake from Tank 25F provided to Interim Salt Disposition Projects for testing
- Actinide Removal Process
- Modular Caustic-side Solvent Extraction Unit

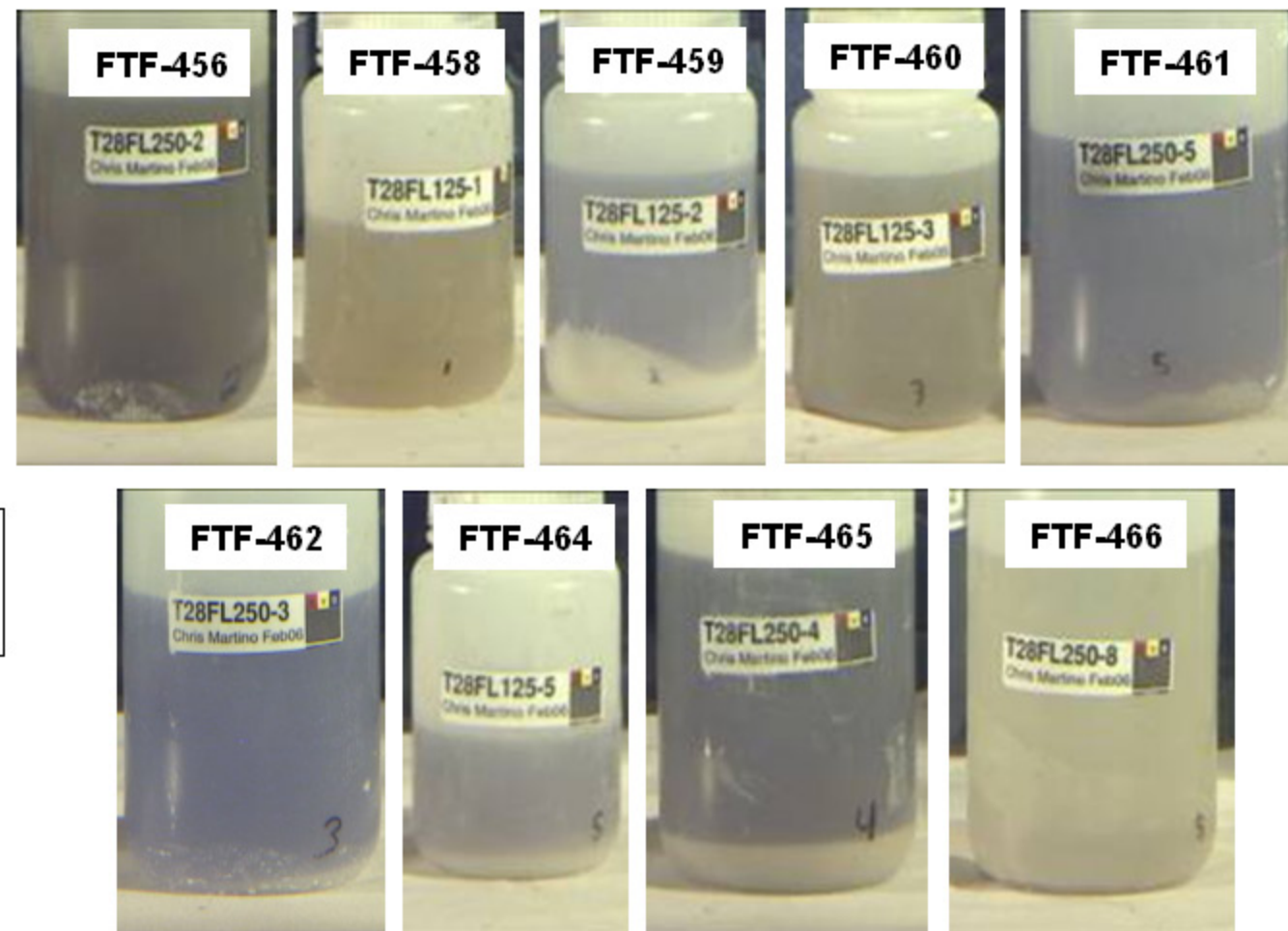




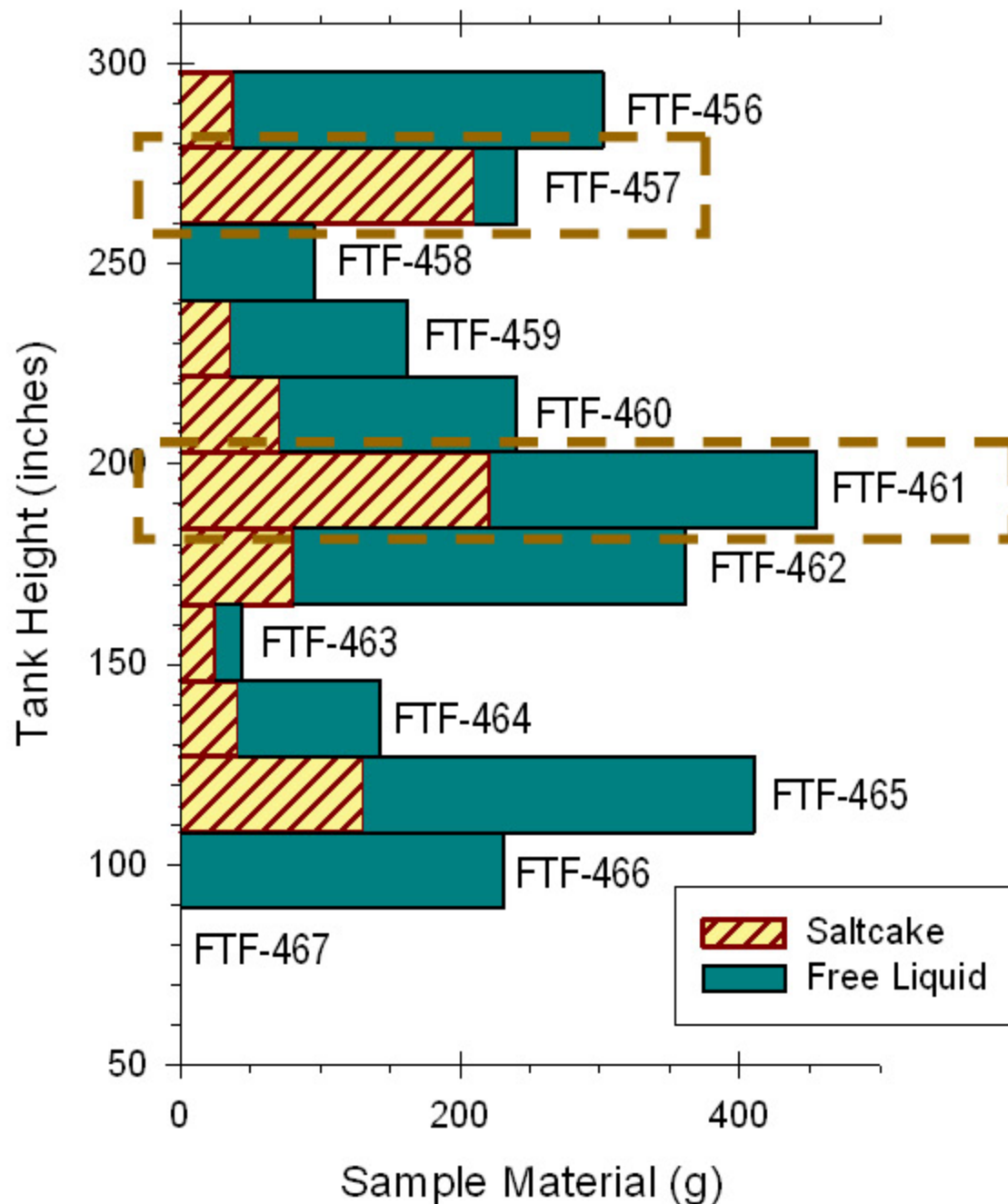
# Tank 28F Saltcake Core Sample Profile



- **Very high Free Liquid content**
  - Interstitial Liquid (IL) contaminated with Drill-String Fluid (DSF)
  - DSF and dissolved salt



# Tank 28F Saltcake Core Sample Profile

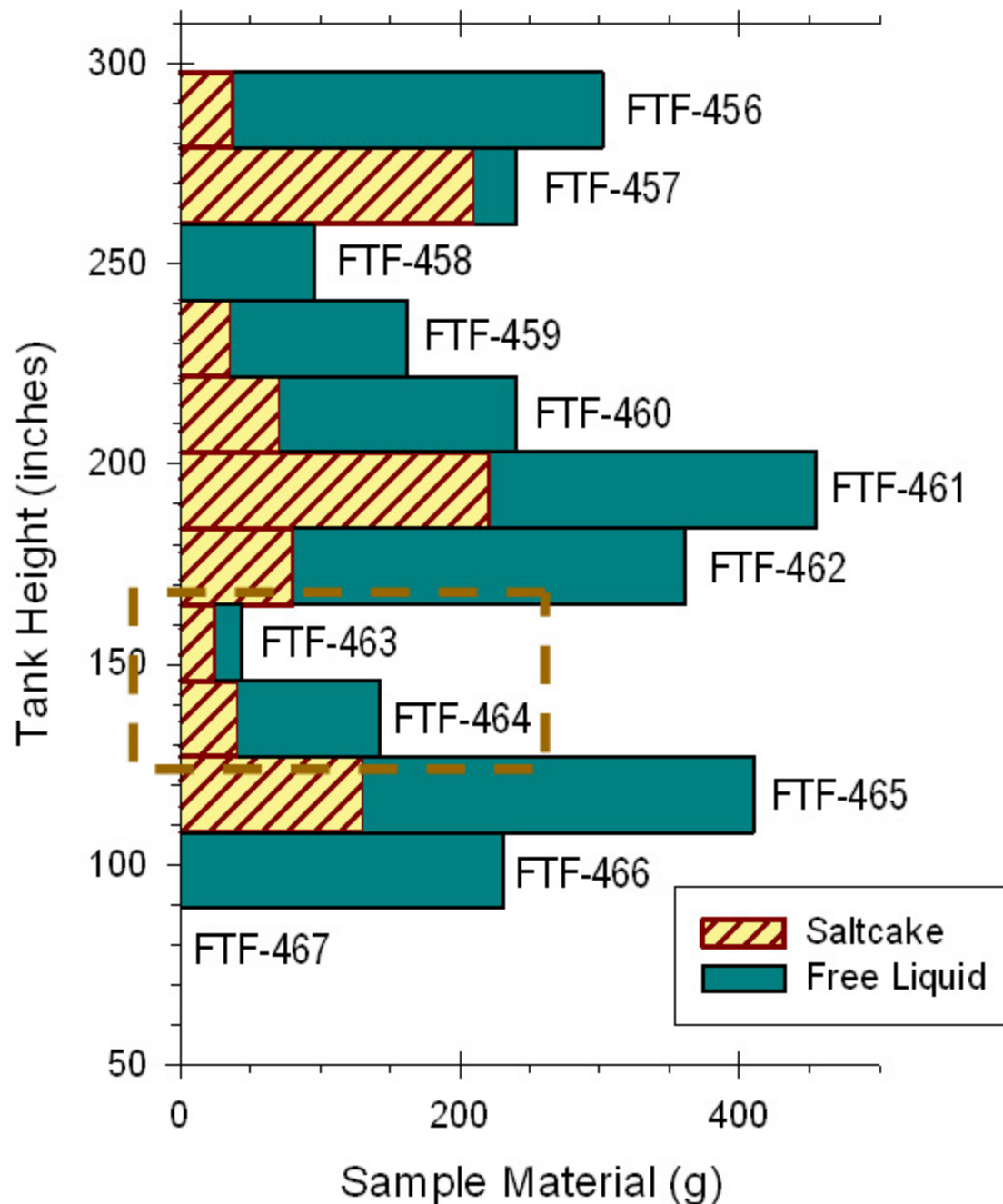


- **Very high Free Liquid content**
  - Interstitial Liquid (IL) contaminated with Drill-String Fluid (DSF)
  - DSF and dissolved salt
- **All saltcake cores were moist**
  - Two good segments obtained
  - Questionable in-tank conditions

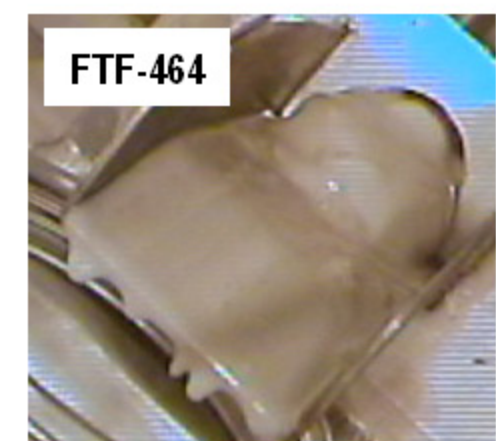
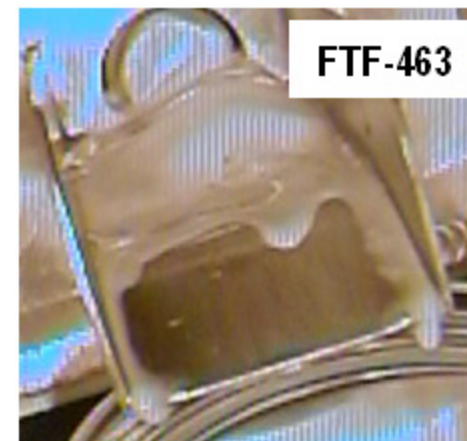




# Tank 28F Saltcake Core Sample Profile

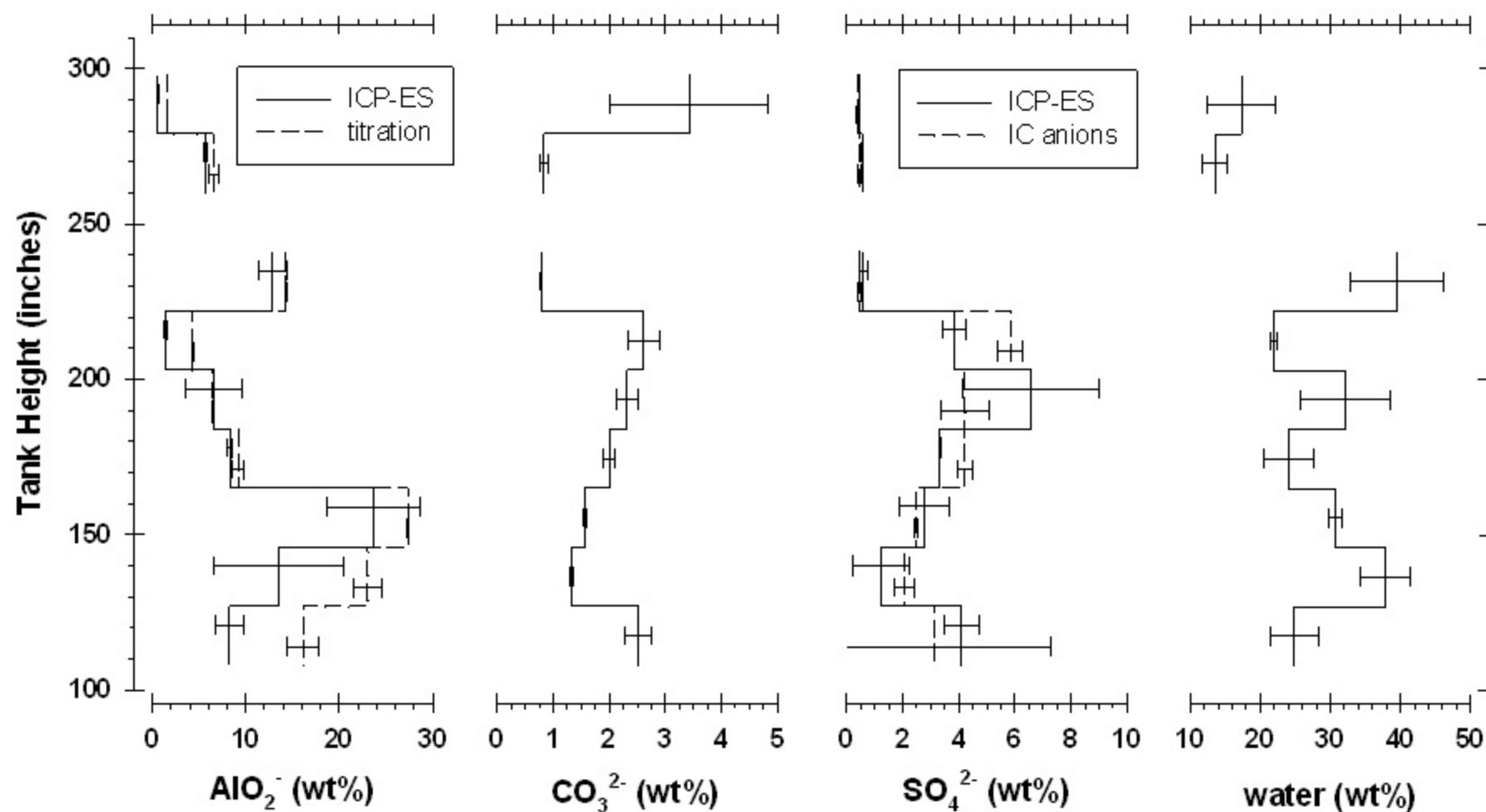


- **Very high Free Liquid content**
  - Interstitial Liquid (IL) contaminated with Drill-String Fluid (DSF)
  - DSF and dissolved salt
- **All saltcake cores were moist**
  - Two good segments obtained
  - Questionable in-tank conditions
- **Two seg. w/ fine white solids**



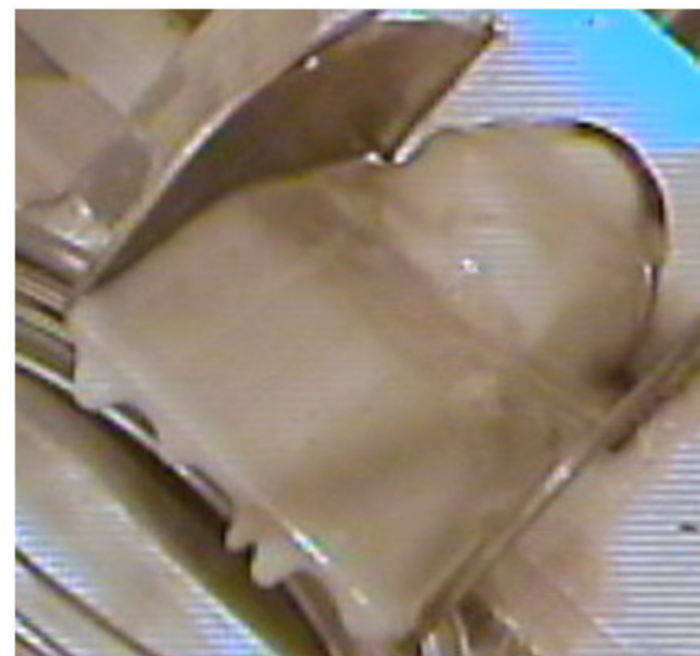
# Tank 28F Results Summary

- Sodium nitrate with sulfate, carbonate, and aluminum
  - Interstitial liquid Cs-137 = 5.0 Ci/gal
  - Saltcake Cs-137 = 1.23 Ci/gal



# Tank 28F Results Summary

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  - High aluminum: 13 to 28 wt% on  $\text{AlO}_2$  basis



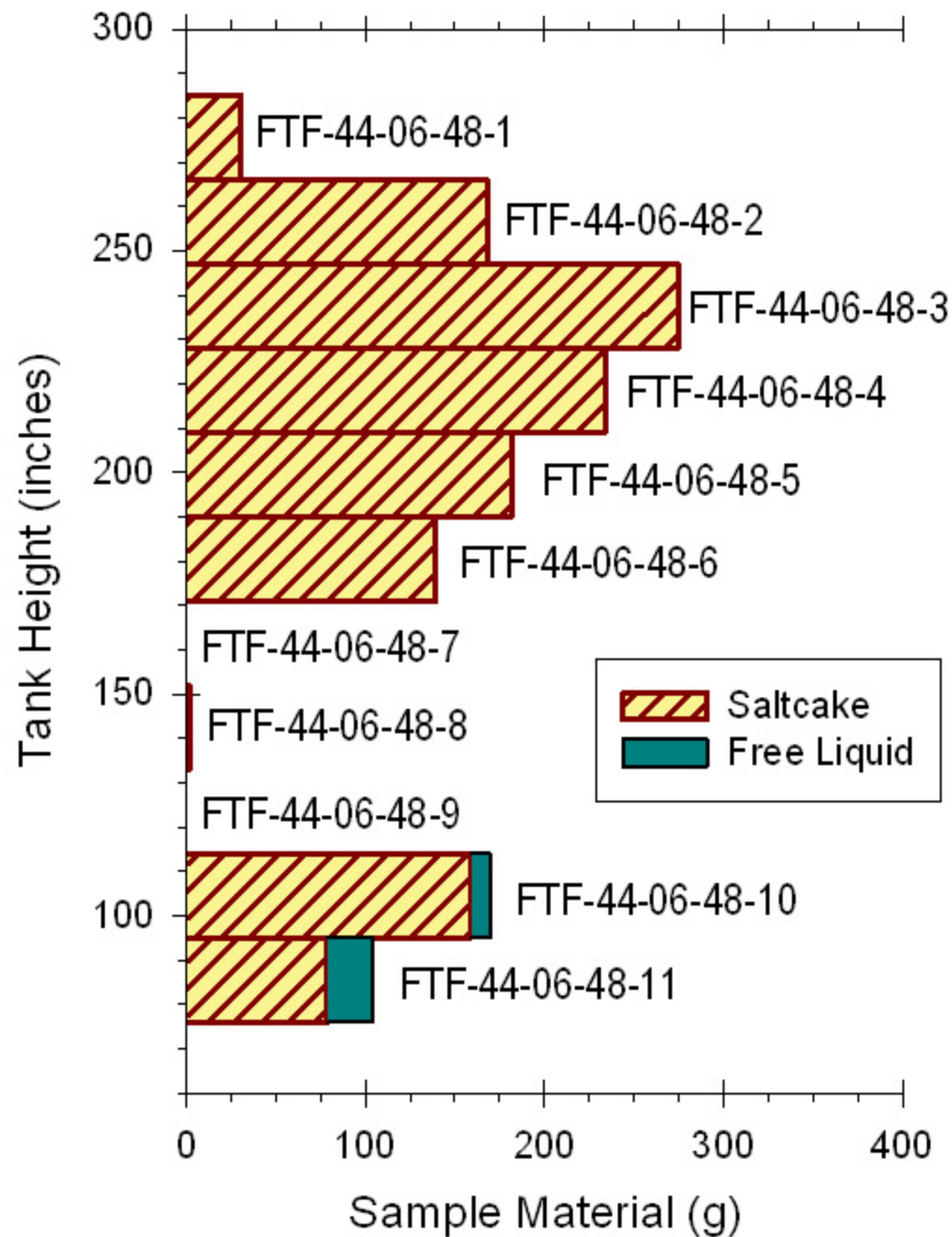


# Tank 28F Results Summary

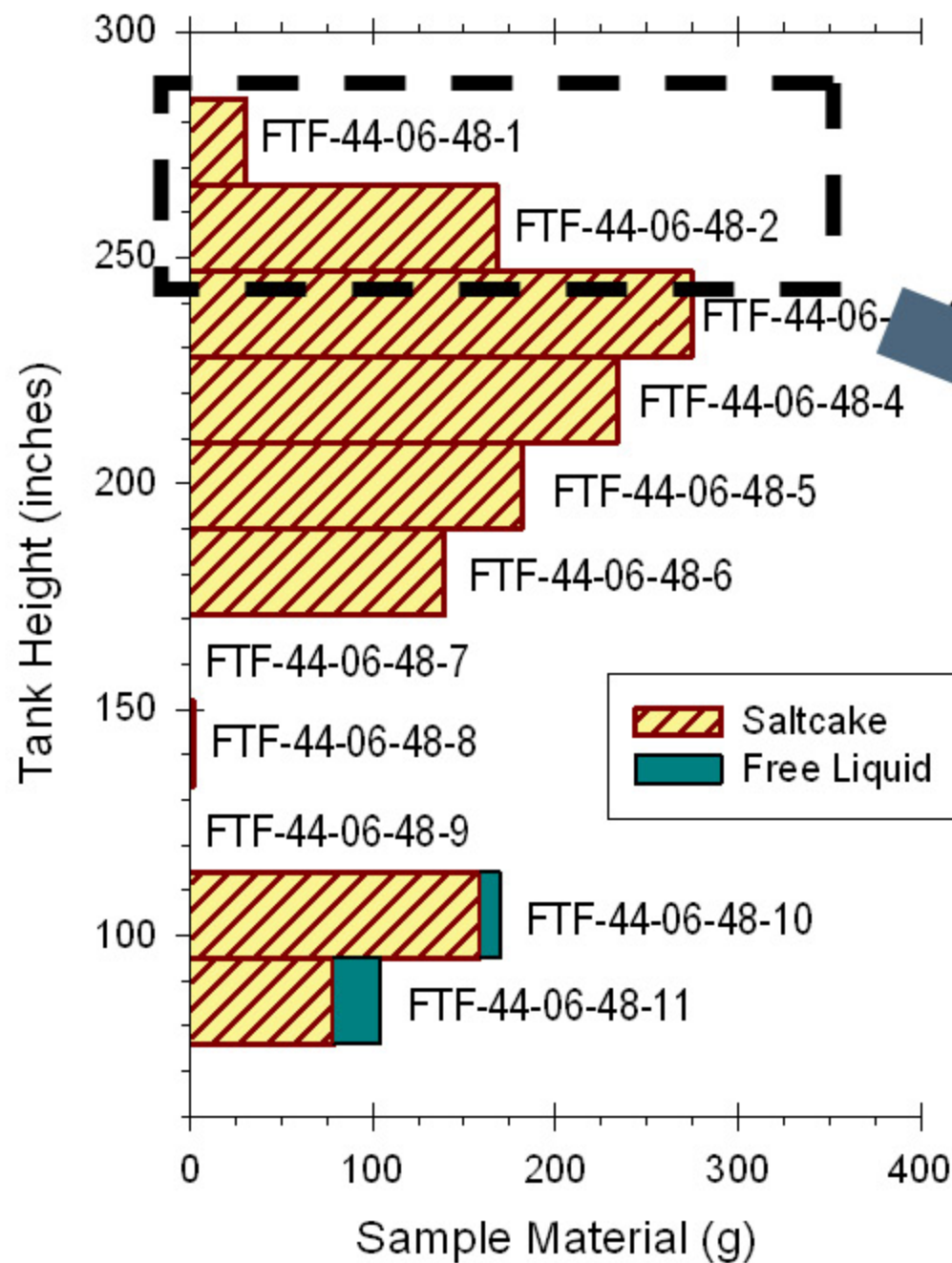
- **Sodium nitrate with sulfate, carbonate, and aluminum**
  - Interstitial liquid Cs-137 = 5.0 Ci/gal
  - Saltcake Cs-137 = 1.23 Ci/gal
- **Two samples with fine white solids**
  - High aluminum: 13 to 28 wt% on  $\text{AlO}_2$  basis
- **Segment-to-segment correlation of soluble components**
  - Moisture content, Cs-137, Tc-99, nitrite, hydroxide, phosphate
- **Single batch dissolution compared w/ Saltstone WAC**
- **Residual insoluble solids – sludge and  $\text{Al}(\text{OH})_3$**

# Tank 44F Saltcake Core Sample Profile

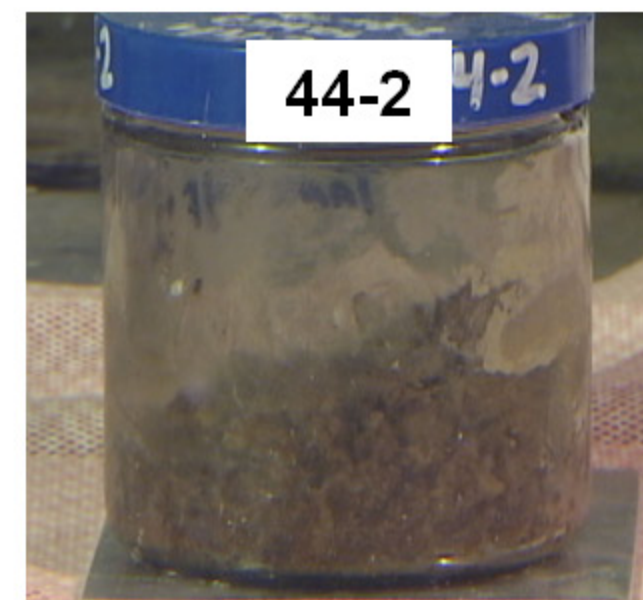
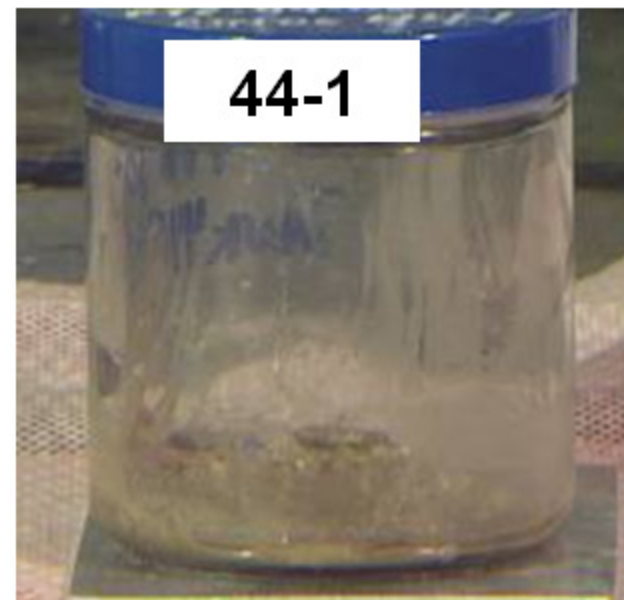
- Relatively little Free Liquid



# Tank 44F Saltcake Core Sample Profile

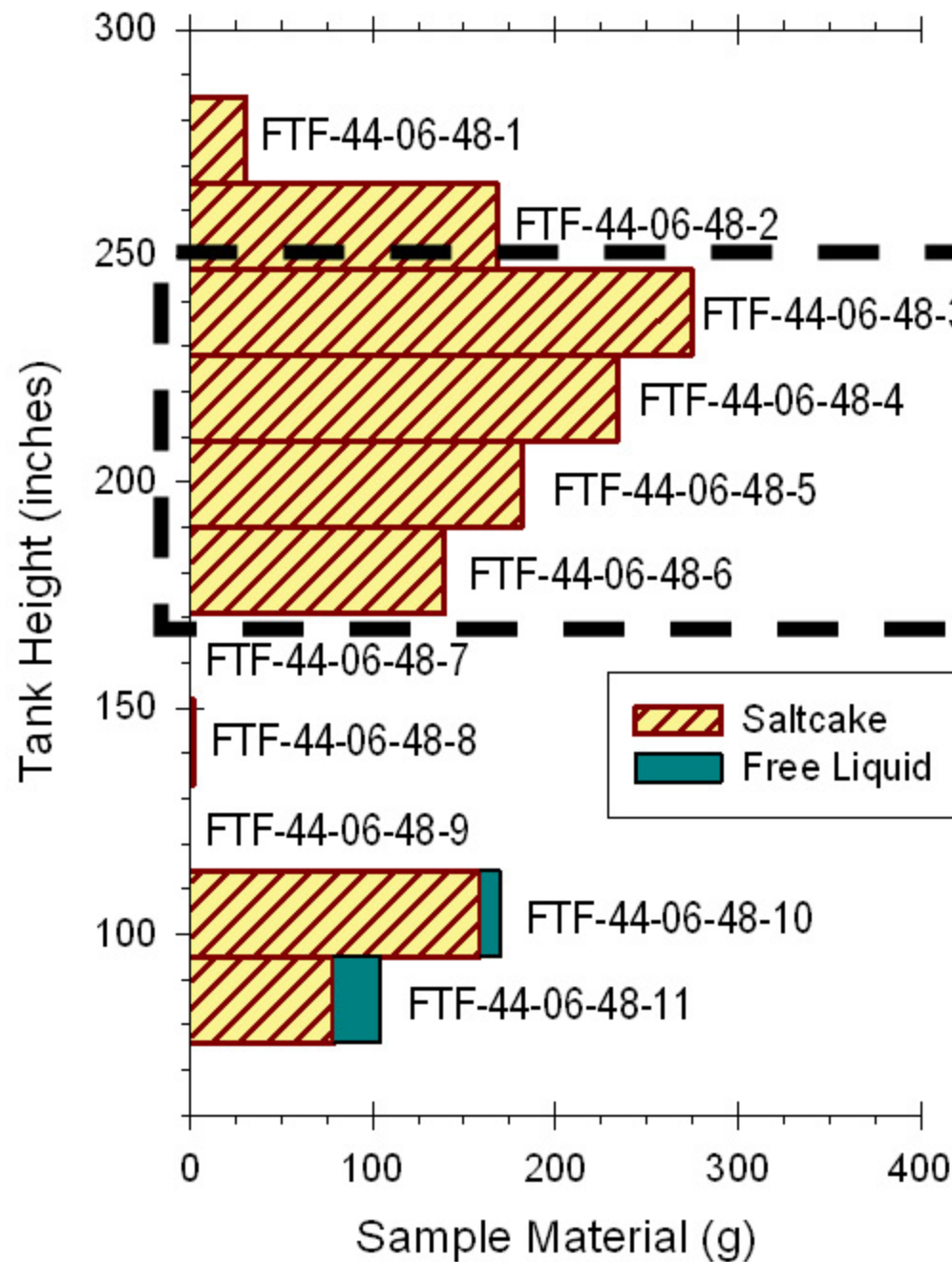


- Relatively little Free Liquid
- Top two samples moist and red shaded

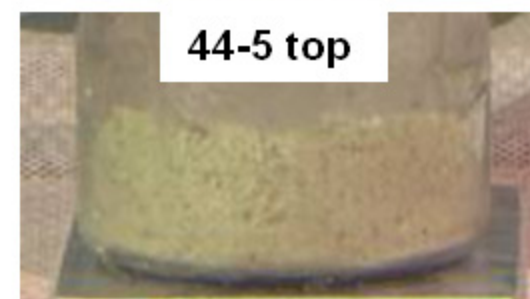
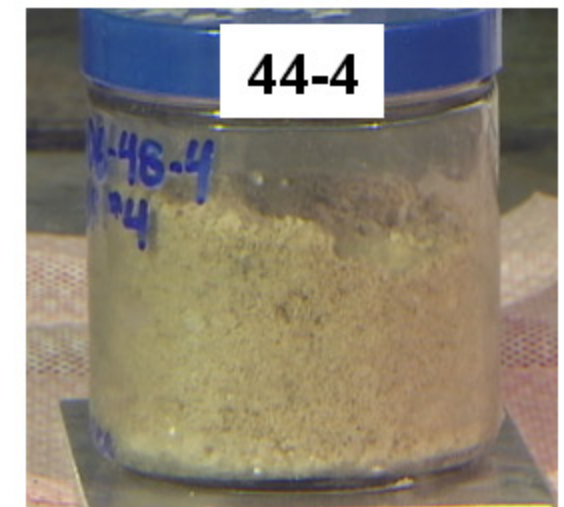
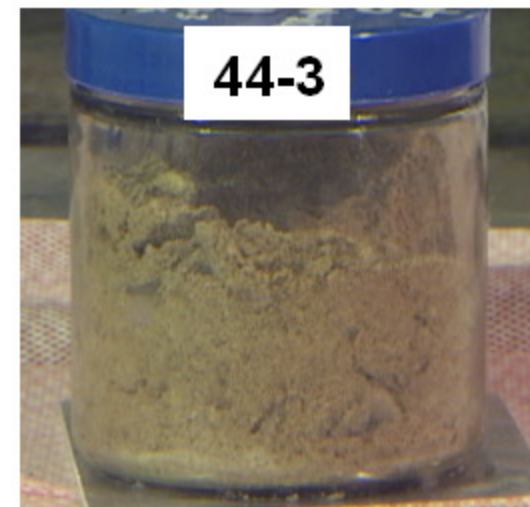




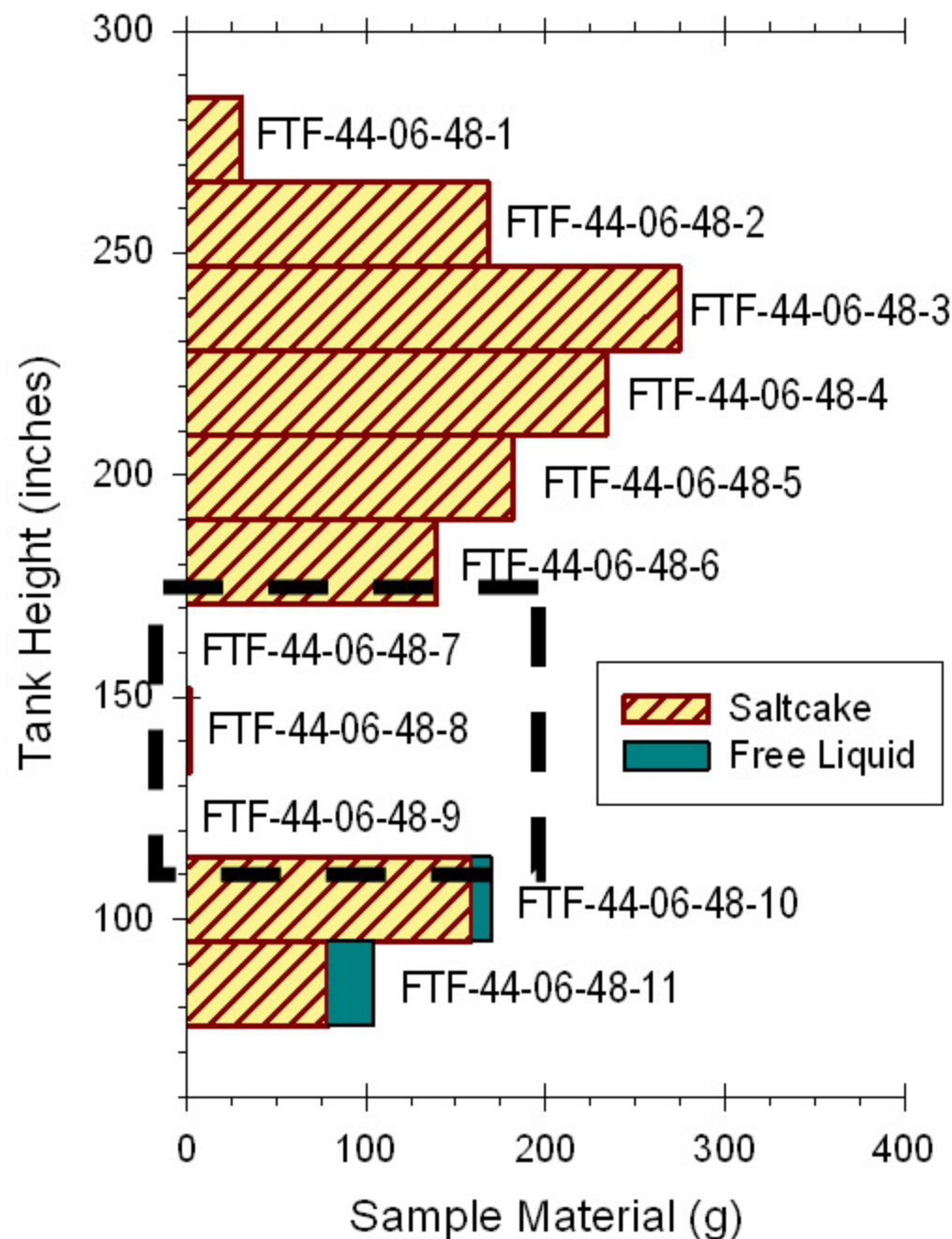
# Tank 44F Saltcake Core Sample Profile



- Relatively little Free Liquid
- Top two samples moist and red shaded
- Next four samples drier and very hard



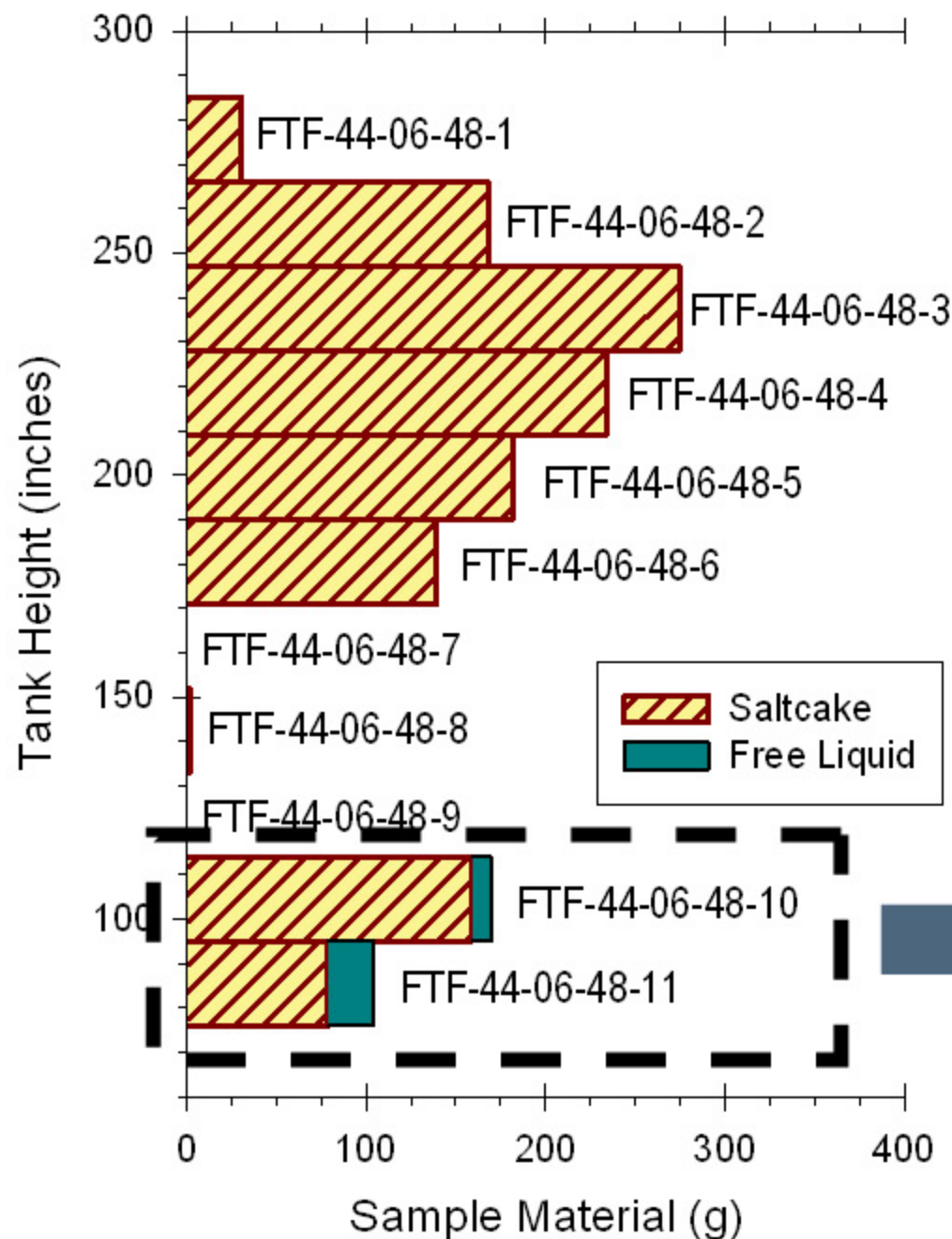
# Tank 44F Saltcake Core Sample Profile



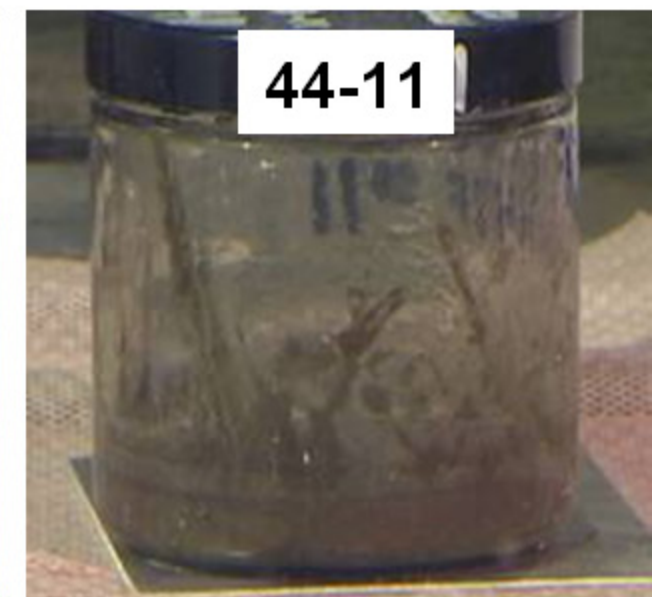
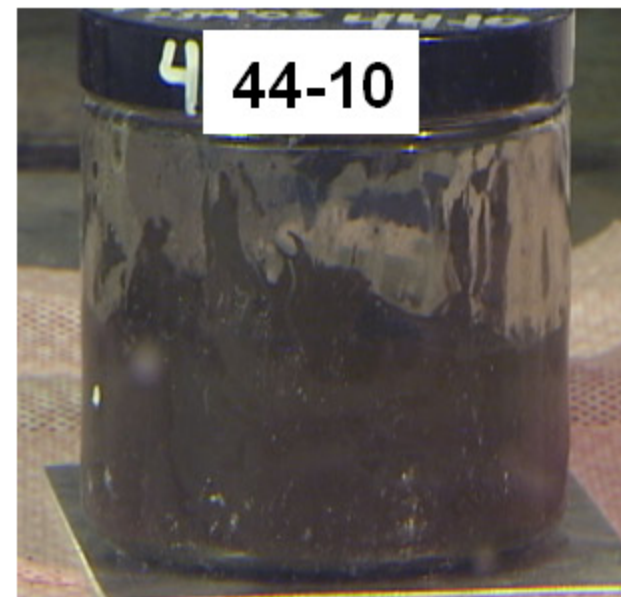
- Relatively little Free Liquid
- Top two samples moist and red shaded
- Next four samples drier and very hard
- Three empty samplers



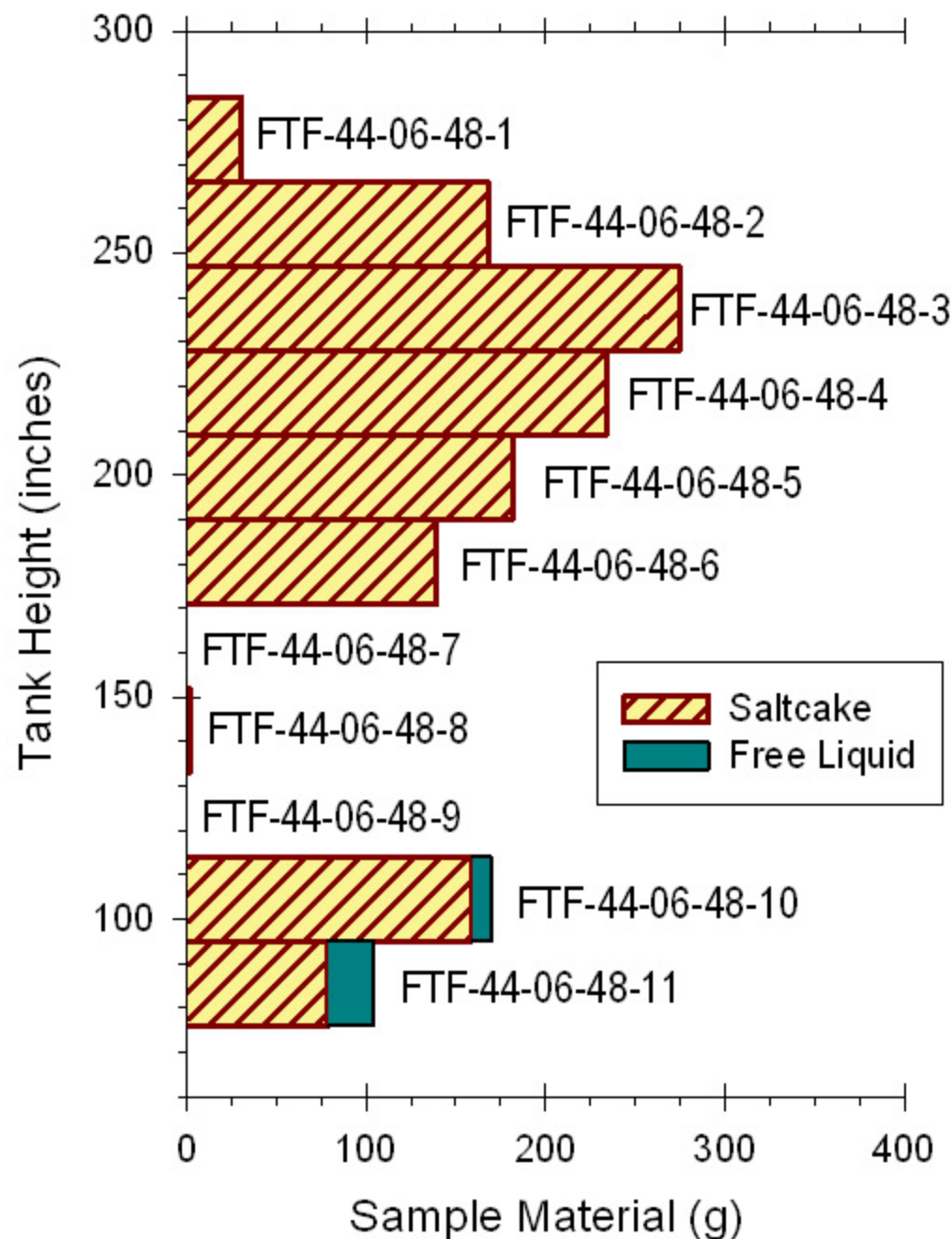
# Tank 44F Saltcake Core Sample Profile



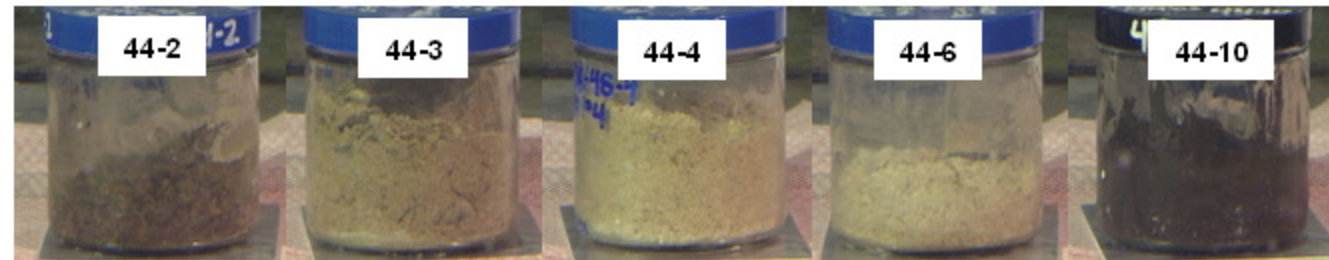
- Relatively little Free Liquid
- Top two samples moist and red shaded
- Next four samples drier and very hard
- Three empty samplers
- Bottom two samples wet and very dark



# Tank 44F Saltcake Core Sample Profile



- Relatively little Free Liquid
- Top two samples moist and red shaded
- Next four samples drier and very hard
- Three empty samplers
- Bottom two samples wet and very dark
- Obvious variety in sample appearance



- Moisture content quantified
- Free Liquid Cs-137  $\approx 4.9$  Ci/gal
- Saltcake held for future characterization

# Summary

- **Tank 25F:** full draining and dissolution test
  - **Segment Analysis** for vertical variation and analyte correlations
  - **Composite Draining** for Interstitial Liquid removal
  - **Dissolution Test** permitted analysis of three batches and heel
    - *Utilized in testing of downstream treatment processes*
- **Tank 28F:** update from previous technical exchange
  - **Segment Analysis** of wet samples showed correlation of solubles
    - *High aluminum content, very moist material*
- **Tank 44F:** received for future processing
  - **Visual Inspection** showed obvious variation → **layering**

# Questions ???

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